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REVISED SHORELINE AND SEDIMENTS INVESTIGATION WORK PLAN

Former Rhone-Poulenc Site

Tukwila, Washington

8A(5)

Prepared for:

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Kent, Washington

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June 2011

Project No. 0087690050.00005

On behalf of the respondents, I certify under penalty of law that this document and all attachments were prepared under my direction or supervision in accordance with a system designed to evaluate the information submitted. I certify that the information contained in or accompanying this Revised Shoreline and Sediments Investigation Work Plan, is true, accurate, and complete. As to those portions of the report for which I cannot personally verify accuracy, I certify under penalty of law that this report and all attachments were prepared in accordance with procedures designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who may manage the system, or those directly responsible for gathering the information, the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations.

By: _____ Date: June 14, 2011

Mr. Gary Dupuy, Project Coordinator

REVISED SHORELINE AND SEDIMENTS INVESTIGATION WORK PLAN
Former Rhone-Poulenc Site
Tukwila, Washington

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Project 0087690050.00005

This report was prepared by the staff of AMEC Geomatrix, Inc., under the supervision of the Hydrogeologist whose seal and signature appear hereon.

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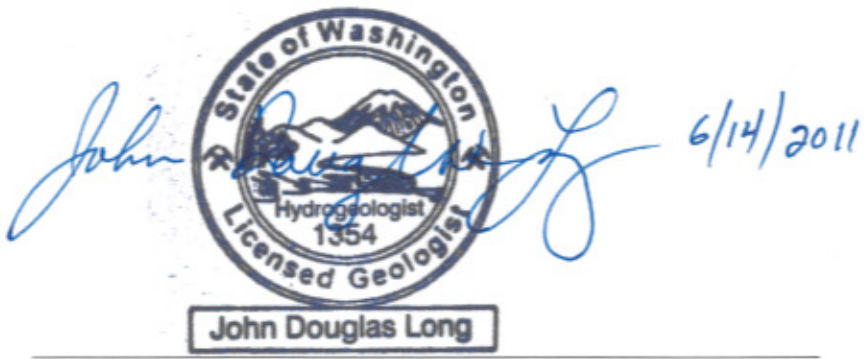

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1.0 INTRODUCTION

AMEC Geomatrix, Inc. (AMEC) has prepared this work plan on behalf of the Respondents in response to a request from the United States Environmental Protection Agency (EPA) for additional characterization of the nature and extent of contamination at the Former Rhone-Poulenc East Marginal Way Facility (site).

1.1 BACKGROUND

The former Rhone-Poulenc facility is located along the Duwamish Waterway (or Duwamish River) at 9229 East Marginal Way South, Tukwila, Washington (Figures 1 and 2). Corrective actions at the site are currently being conducted under Administrative Order on Consent No. 1091-11-20-3008(h) (Order) and are being overseen directly by the EPA.

Industrial use of the site began in the 1930s when I.F. Laucks built a pilot plant to formulate glue for use in plywood manufacturing. In 1949, Monsanto Chemical Company (Monsanto) purchased the site and continued the manufacture of glue, as well as paints, resins, and wood preservatives. Monsanto began vanillin production in 1952 which continued until the sale of the property to Rhone-Poulenc in 1985. Because the facility stored hazardous waste, it was subject to the requirements of the Resource Conservation and Recovery Act (RCRA), and Monsanto submitted notice of application for permitting under RCRA in the form of a RCRA Part A Interim Status Permit Application. The site is under RCRA interim status, and site environmental issues are regulated under RCRA. Rhone-Poulenc closed the site permanently in April 1991 and transferred the title of the property to Rhodia, Inc. (Rhodia) in January 1998. Rhodia sold the property on July 13, 1998, to Container Properties L.L.C., the current owner. Container Properties' intent is to clean up the site and redevelop it as industrial property.

Since site closure in 1991, extensive investigations have been completed at the site to evaluate environmental impacts to soil and groundwater from the former manufacturing plant. The investigations have followed the RCRA process from an initial RCRA Facility Assessment (RFA) (PRC, 1990) through a 1991 Site Assessment (Landau, 1991), a RCRA Facility Investigation (RFI) (CH2M HILL, 1995), and a Risk Assessment/Media Cleanup Standards Report (RA/MCS) (AGI Technologies, 1999). Studies completed subsequent to the RFI include Geoprobe® and geotechnical investigations conducted in support of interim measure design (URS, 2002a) and a

Geoprobe investigation (AGI Technologies, 2001). The two Geoprobe investigations performed by AGI involved collection of groundwater samples from the western and southern sides of the property. Groundwater samples were collected from these Geoprobes at depths of 15 (if possible), 20, 25, 30, 35, 40, 45, and 50 feet. These groundwater samples were analyzed for metals and pH. Approximately 50 percent of the Geoprobes were installed within 50 feet of the Duwamish River or Slip 6 shorelines. Figure 2 shows the location of these Geoprobe samples. The sample results showed that shoreline areas contained elevated concentrations of metals, including copper, arsenic, and mercury, in addition to elevated pH readings.

Prior to redevelopment of the site in 2006, the following additional investigations of historical structures and buildings, potential waste disposal areas, and sumps uncovered during redevelopment were conducted, as shown in Figure 3:

- Pre-Demolition Investigation: Investigation of buried facility structures, sumps, and basements prior to redevelopment of the site (Geomatrix, 2006a). Elevated concentrations of metals, semivolatile organic compounds (SVOCs), polychlorinated biphenyls (PCBs), and toluene were detected in samples collected from the former Scale Pit in the main warehouse, the Copper Sump, and the 1-120 Sump. All liquids and sediment were removed from these structures, stabilized, and disposed of in accordance with state and federal regulations.
- Hazardous Waste Storage Area (HWSA) and Transformer A Cleanups: During redevelopment and regrading of the site soil contamination associated with a buried catch basin in the closed hazardous waste storage area and leakage from a former electrical transformer (Geomatrix, 2006b).
  - Soil near the former HWSA catch basin contained total petroleum hydrocarbons (TPH), SVOCs, and metals. The affected soil around the catch basin was removed, and the catch basin was pumped out and abandoned in place.
  - A former electrical transformer had leaked and contaminated the underlying soil with TPH-diesel (TPH-D). Soil samples indicated that PCB concentrations in the soil were below 1 mg/kg. The transformer was removed and 36 tons of TPH-D-affected soil around the transformer location was excavated and disposed of off site at the Roosevelt landfill.
- Former Oil/Water Separator Investigation: Investigation and removal of water and sediments under a former oil/water separator uncovered during regrading of the site (Geomatrix, 2006c). The oil/water separator was drained of all liquids and sediment, steam-cleaned, and abandoned in-place. All liquids removed from the structure were stabilized and disposed of and treated off site.
- East Parcel Soil Characterization and Voluntary Interim Measure: Areas of the former East Parcel were investigated for possible contaminated soil. Areas of contaminated soil were excavated and removed from the East Parcel prior to the subdivision of the site later in 2006 (Geomatrix, 2006d).

- Northwest Corner Soil Removal: An area in the northwest corner of the facility outside of the barrier wall (see the paragraph below discussing the hydraulic control interim measure [HCIM]) was characterized for copper, TPH-gasoline (TPH-G), and TPH-D, although the TPH-G detection was characterized by the laboratory as "mineral spirits" rather than gasoline. Approximately half of the copper-affected soil with lower concentrations was excavated and placed in the contained area within the barrier wall. The remaining soil with higher concentrations of copper or TPH-G was disposed of off site in accordance with state and federal regulations (Geomatrix, 2007).

The primary constituents of concern (COCs) for the site are:

- Toluene, an industrial solvent used in the vanillin manufacturing process;
- Copper in soils and groundwater resulting from vanillin black liquor solids used for weed control, various releases of contaminated surface runoff waters and process waste waters, and strainer solids from vanillin manufacture; and
- Groundwater affected by elevated pH due to caustic releases.

Toluene-affected groundwater is limited primarily to the southwest portion of the site. Copper-affected groundwater and groundwater having elevated pH due to the caustic release are limited to the southwestern corner of the site, based on historical data. Other COCs for the site include polycyclic aromatic hydrocarbons (PAHs), methylene chloride, benzene, arsenic, chromium, lead, mercury, nickel, and vanadium. In addition, SVOCs, including pentachlorophenol, have been documented at the site.

Elevated concentrations of PCBs have also been observed in an area affected by past releases from a former PCB-containing compressor. PCB-contaminated soils around the compressor pad and a decommissioned underground drain line were removed during two separate interim measures (Rhodia, 1998; Geomatrix 2006e). Sources of metals (such as the use of metals sludge for weed control or the burial of autoclave solids) and other contaminants are described in the RFI report (CH2M HILL, 1995).

The interim groundwater remedy used at this site is hydraulic containment. An HCIM was constructed at the site from January through July 2003, consistent with the EPA-approved work plan (URS, 2002b). The HCIM consists of a low-permeability, subsurface barrier wall with a groundwater extraction and treatment system designed to maintain an inward-directed groundwater gradient. The extracted groundwater is treated using granular activated carbon (GAC) and discharged to a publicly owned treatment works (POTW). The HCIM is monitored with a network of monitoring wells with a monitoring program designed to evaluate chemical constituents in groundwater and water levels within and outside of the HCIM area.

The ongoing groundwater monitoring performed at the site since 2003 shows that:

- Toluene and copper concentrations in groundwater samples from the western downgradient side of the barrier wall have decreased or stabilized at lower concentrations since completion of the HCIM.
- Copper and toluene concentrations and caustic pH readings in the southwestern exterior of the HCIM barrier wall have stabilized at higher concentrations in groundwater samples collected from three of the performance monitoring wells; copper is present at concentrations ranging from 50 to 200 micrograms per liter (µg/L); toluene concentrations remain elevated, and pH readings exceeding 9.5 pH units have also been recorded from these same wells.

In the last 8 years of groundwater monitoring, concentration trends in copper, toluene, and pH have varied over time in samples collected from these wells, with no overall decreasing or increasing trend. While not part of the ongoing groundwater monitoring, it should be also be noted that other constituents have been found at the site, including PAHs, PCBs, phthalates, pentachlorophenol, and various hydrocarbons.

In 2006, the entire facility underwent redevelopment, and additional subsurface investigations were performed. The property was split into two parcels, the East Parcel and the West Parcel. The East Parcel was extensively investigated and remediated. EPA provided a partial determination of "Corrective Action Complete without Controls" for the East Parcel in a letter dated December 20, 2006 (EPA, 2006). The partial determination was made since a portion of the property, approximately 2,000 square feet in size in the extreme southwestern corner of the East Parcel, was found to have soil and groundwater impacted with toluene above project-specific cleanup goals. Corrective actions were undertaken for this portion of the property using combinations of air sparge, biovent, and/or soil vapor extraction systems to treat toluene in the soil and groundwater. Some combination of these systems was operated from December 2008 until June 2010, when the systems were shut down. Container Properties is continuing to monitor groundwater quality in this area to confirm that corrective actions are complete. The East Parcel is now owned by the Museum of Flight, and throughout this work plan the former East Parcel will be referred to as the Museum of Flight Property.

The West Parcel was regraded and repaved as part of redevelopment activities. The West Parcel is now leased by Container Properties to International Auto Auctions, Inc. (IAAI). This work plan applies to investigation activities associated with the former West Parcel, which will be referred as the IAAI Lease Property, the former Rhone-Poulenc facility, or the site.

The HCIM has been in operation at the site since August 2003, and continues controlling migration of groundwater within the barrier wall. However, the subsurface barrier wall is set back from the Duwamish Waterway approximately 50 feet, and therefore soil and groundwater outside the wall are

not controlled or captured. Just prior to redevelopment, limited excavation of soils affected by copper and petroleum hydrocarbons was completed in the northwest corner of the IAAI lease property in an area just outside the barrier wall (see Figure 2 for location). Soil sampling was conducted in the northwest corner, but the nature and extent of copper- and petroleum-affected soil was not determined to the south of northwest corner sampling location 42. Based on prior investigations and the on-going groundwater monitoring, copper-affected soil and groundwater are known to be present in the southwest corner along the shorelines of the Duwamish River and Slip 6.

The *Lower Duwamish Waterway Group Feasibility Study* (AECOM 2010) was reviewed for sediment analytical results collected adjacent to the project site. The sediment sample results from the EPA sediment sampling investigation in 2004 are also included in the overall dataset (EPA, 2005). Sediment samples from the dataset include surface sediment samples (collected at depth interval 0 to 0.33 foot or 0 to 10 centimeters [cm]) that were collected from 97 surface sample locations. The dataset also includes subsurface samples (0.33 foot in depth or greater) that were collected at 25 locations. Some of the samples were analyzed for the complete Sediment Management Standards (SMS) list of COCs. Additional samples were analyzed for limited lists of analytes, including additional metals, pesticides, PCB by congeners, dioxins and furans, volatile organic compounds (VOCs), miscellaneous SVOCs, and organometallic compounds.

Table 1 presents a summary of the chemistry results for the SMS chemicals, pesticides, and additional metals. The total number of samples analyzed for each chemical parameter is presented along with the number of samples in which the parameter was not detected versus the number of samples in which the parameter was detected. The detection frequency is calculated using the counts. The maximum detected concentration of a chemical parameter at any location is also shown in Table 1. Table 1 also shows the number of samples with detected concentrations that exceeded the associated Sediment Quality Standard (SQS) criterion and Cleanup Screening Level (CSL) criterion for analytes with an established SMS criterion. Analytical results for one or more samples exceeded either the SQS or the CSL criteria for the following analytes: mercury, acenaphthylene, benzoic acid, phenanthrene, total high-molecular-weight PAHs (HPAHs), indeno(1,2,3-cd)pyrene, dibenzo(a,h)anthracene, benzo(g,h,i)perylene, bis(2-ethylhexyl)phthalate, diethyl phthalate, di-n-octyl phthalate, phenol, pentachlorophenol, benzoic acid, dibenzofuran, and total PCBs.

In a letter dated April 28, 2009, EPA requested that additional investigation be completed in three areas of the former Rhone-Poulenc facility: the Slip 6 bank, the Duwamish riverbank, and the sediments in the offshore area (EPA, 2009). The Respondents, including Container Properties, requested a meeting with EPA to negotiate options regarding completion of the additional work. Due to several scheduling conflicts, this meeting was held at EPA Region 10 offices on August 12, 2010.

EPA subsequently sent a letter to the Respondents dated August 18, 2010, indicating that the additional work EPA requested is still required and that the Respondents should submit a work plan for the additional work by October 18, 2010 (EPA, 2010a). The Respondents requested an extension to the October 18, 2010, deadline for submittal of the work plan (AMEC, 2010). In its reply dated September 16, 2010, EPA approved the Respondents' request for an extension of the deadline for submittal of the work plan to November 19, 2010 (EPA, 2010b). This work plan has been prepared in response to EPA's request for additional work.

1.2 PURPOSE

The purpose of additional investigation of the shorelines and sediments of the IAAI Lease Property is to define the nature and extent of COCs in soil, groundwater, pore water, and/or sediments remaining on the site outside of the downgradient sides of the barrier wall. The investigation will involve:

- Determining the nature and extent of metals, volatile organic compounds (VOCs), and elevated pH in soil, groundwater, and pore water along the southwest and the Slip 6 sides of the HCIM barrier wall;
- Determining the nature and extent of metals, VOCs, SVOCs, PCBs, and TPH in soils along the Duwamish riverbank west of the HCIM barrier wall; and,
- Determining the nature and extent of the Sediment Management Standards (WAC-173-204) COCs, as well as dieldrin and vanadium, in the tideflat sediments and extending into the channel and Slip 6 as necessary to fully delineate the nature and extent of contaminants located at or released from the facility.

The sediment investigation may also include the collection of intertidal sediments at selected locations within Slip 6 adjacent to the IAAI Lease Property. Access agreements with the adjoining property owners will be pursued. Determination of the nature and extent of COCs in the various media investigated will be compared to relevant environmental standards including:

- Cleanup levels for the Duwamish River that may be established by EPA prior to completion of the Corrective Measure Study (CMS).
- Regional Screening Levels for chemicals developed by EPA Region 9.
- Washington State Department of Ecology (Ecology) Model Toxics Control Act (MTCA) Method A cleanup levels for soil and groundwater
- Ecology MTCA Standard Method B cleanup levels (carcinogen and non-carcinogen) for soil and groundwater.
- Puget Sound Region natural background concentrations for metals in soil.
- Surface water quality standards for fresh water established under Ecology's WAC-173-201A, the National Toxics Rule in 40 Code of Federal Regulations part 141, and Section 304 of the Clean Water Act.

- Ecology's MTCA Method B surface water quality standards (carcinogen and non-carcinogen).
- SQS and CSLs established under the Washington State Department of Ecology's Sediment Management Standards.

The exact environmental standards will be determined during the CMS, which will be conducted when the shoreline and sediment investigations are completed. The location, depth, and type of contamination identified during the shoreline and sediment investigations will determine which cleanup actions are evaluated and selected in the CMS for the Slip 6 and southwest shoreline areas, the Duwamish River shoreline areas, and the tideflats.

1.3 PROJECT ORGANIZATION

An organizational chart showing lines of authority and reporting responsibilities is presented on Figure 4. AMEC is the prime consultant working under contract to Container Properties, L.L.C. Gary Dupuy of AMEC is the Respondent's Project Coordinator. AMEC's project manager for the Shoreline and Sediment Investigation is John Long. He will be responsible for overall supervision of the work described in this work plan. The remaining personnel on the project team and their roles are listed on Figure 4. A more detailed description of individual roles and responsibilities is presented in the project-specific Quality Assurance Project Plan (QAPP) in Appendix A.

1.4 ORGANIZATION OF WORK PLAN

This work plan consists of the following sections:

Section 1.0 Introduction: Describes the purpose of the work plan, provides background information, and describes the project objectives and organization;

Section 2.0 Shoreline Sampling and Analysis Plan: Describes the methodology and procedures for carrying out the shoreline investigation, which involves sampling between the barrier wall and the top of the bank;

Section 3.0 Sediment Sampling and Analysis Plan: Describes the methodology and procedures for carrying out the sediments investigation;

Section 4.0 Deliverables: Specifies the documents and reports to be submitted for conducting and reporting on the investigation;

Section 5.0 Schedule: Specifies the schedule and timing of the investigations;

Section 6.0 Health and Safety: Specifies the procedures to be followed to protect worker health and safety while conducting the investigation;

Section 7.0 References: Presents a list of references cited in the work plan.

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2.0 SHORELINE SAMPLING AND ANALYSIS PLAN

Samples of soil and/or groundwater will be collected along the shoreline in accordance with the sampling and analysis plan presented here. This plan was developed following the EPA's *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA, Interim Final* (EPA, 1988). The shoreline sampling will be conducted in accordance with the existing site-specific health and safety plan that was developed for use during past soil and groundwater sampling events (AMEC, 2009).

Figure 5 shows the proposed shoreline sampling locations for soil and/or groundwater along the southwestern corner of the site, along Slip 6, and along the Duwamish riverbank. Samples along the southern property boundary will be collected between the barrier wall and the electrified fence installed by IAAI that is adjacent to the property boundary. Samples along the western property line will be collected between the electrified fence and the older facility fence.

2.1 SAMPLING OBJECTIVES

Results from sampling conducted outside and downgradient of the barrier wall, in addition to historical data collected from previous investigations at the facility, will be used to conduct a CMS as required by Section VI.D and Attachment C of the Order. Additional data may be necessary to complete final designs, depending on the complexity of the selected corrective measures. This additional data may be collected during or prior to implementation of the selected corrective measure alternative.

The data quality objectives of the shoreline and sediment investigations are to:

- Determine the nature and extent of site COCs in soil, groundwater, pore water, and sediments along the downgradient side of the barrier wall.
- Compare the detected concentrations of COCs to the relevant environmental quality criteria applicable to these media.
- Use the results of this comparison to determine the appropriate corrective measures to address the site COCs that pose a risk to human health and the environment during the CMS.

Table 2 lists the potential environmental criteria and the reporting limits for potential soil and groundwater COCs that may be detected during the investigation. The conceptual site model for this site is presented in Section 2.1.1. This model identifies the potential routes for site COCs to affect soil, groundwater, surface water, and sediments.

2.1.1 Conceptual Site Model

Figure 6 shows a conceptual model for the portion of the site on the downgradient side of the barrier wall along the Duwamish River. The location of the areas of groundwater affected by copper, toluene, and pH are based on the 1999 and 2001 direct-push investigations (AGI 1999, 2001).

Figure 6 shows the former facility layout including the location of the former stormwater conveyance system at the site. Up to seven outfalls at the site were used to route stormwater from the facility into Slip 6 and the Duwamish. It is not known which outfalls were used at different times or when specific outfalls were abandoned. Outfall No. 1 is a King County storm sewer that extends from Boeing Field to Slip 6 and a portion of the former facility where the laboratories and offices were located on the East Parcel.

As shown on the historical facility sewer maps, there were bypasses connecting portions of the process area and process sewer to the stormwater conveyances. Therefore, it is possible that process chemicals could have been routed to the storm sewers at times. AMEC has no knowledge of how these bypasses were used during facility operations, and the facility drawings note that some of these bypasses were abandoned at some time in the past.

There was a barge loading dock used for loading and/or off-loading materials. Figure 6 shows the approximate location of the barge dock and the pier that formerly connected the dock to the facility. It is possible that there were incidental losses of material from barges during transfer of materials to and from the facility; however, AMEC does not have specific knowledge of any specific incidents.

PCBs were likely used on site in electrical transformers and equipment until PCBs were phased out of use in the late 1970s. AMEC has no specific knowledge of the location of the former PCB-containing transformers at the facility. PCBs (Aroclor 1254) were also used as heat transfer fluid in the former "autoclave" building on site. Autoclaves were used to promote catalyzed reactions forming vanillin under heat and pressure (Kong, 2006).

Prior to the construction of the barrier wall, shallow groundwater would flow into Slip 6 and the Duwamish River. Groundwater would discharge into the river due to advective groundwater flow as well as discharging through seeps in the subtidal areas bordering the site.

During construction of the barrier wall, a shallow trench was excavated along the barrier wall path to a depth of 15 to 18 feet both to contain the grout slurry during installation of the barrier wall and to abandon and cap any historical outfalls or pipes crossing the trench path. After construction of the barrier wall, the connection to Outfall No. 7 was replaced, and this outfall was used to route stormwater to the Duwamish from April 2003 until September 2006. In September 2006, the

West Parcel stormwater was reconnected to the King County storm sewer near the southeast corner of the barrier wall through a Contech Stormwater Management "Stormfilter" unit as required by the City of Tukwila (Geomatrix, 2007).

Since construction of the low-permeability barrier wall, groundwater on the downgradient or "lee" side of the barrier wall has likely stagnated because the barrier wall effectively prevents groundwater flow from the upgradient side of the barrier wall. The barrier wall has a design hydraulic conductivity of at least 1.0×10^{-6} centimeters per second and functions as a "no-flow" boundary. Groundwater flow on the lee side of the wall would be influenced by tidal water level fluctuations in the Duwamish River and high water caused by seasonal rainfall and releases from the Howard Hansen Dam. Such periodic changes in water level increase residence time for groundwater on the lee side of the wall. The overall flux of groundwater along the downgradient side of the barrier wall into the Duwamish and Slip 6 should have decreased since installation of the barrier wall.

Since installation of the barrier wall, the pH readings collected from wells DM-8 and MW-42 have decreased to approximately 7 and 7.5 pH units after spiking in 2004. Alkaline material was used to accelerate the curing of the low-permeability grout during installation of the barrier wall. This alkaline material may have contributed to the temporary rise in pH readings.

To summarize:

- Chemicals used and handled on site during past operation of the facility have affected soil, groundwater, and sediments at or near the site.
- Past stormwater discharges from the facility may have introduced COCs into Slip 6 and the Duwamish River channel, where these COCs may have partitioned into sediments.
- Migration of groundwater from the site may have introduced COCs into surface water, and some of these chemicals could have partitioned into sediments.
- Since installation of the barrier wall and operation of the groundwater extraction system began in August 2003, the flux of COCs from the site has decreased; however, COCs remain in the soil and/or groundwater located between the barrier wall and the Duwamish River.
- Redevelopment of the site in 2006 has minimized the amount of surface water infiltration at the site and improved the performance of the groundwater pretreatment system. The majority of the site is now paved and sloped to encourage drainage of stormwater into a new stormwater system.

The data gaps that remain include:

- The nature and extent of COCs in the soil along the Duwamish River shoreline;

- The nature and extent of COCs in the soil, groundwater, and pore water along the southwest side of the barrier wall and the Slip 6 shoreline; and
- The nature and extent of COCs remaining in the sediment in the intertidal area.

2.1.2 Potential for Habitat Restoration and Remedies

The scope of the shoreline and sediment sampling is determined by the likely restoration of habitat along the Duwamish River channel. The intertidal area on the west side of the site is one of the larger remaining intertidal areas along the Duwamish, and restoration of habitat is both desirable and valuable due to off-setting of environmental damages associated with past facility operations. Therefore, it is highly likely that habitat restoration will be a primary component of the remedy for the Duwamish River shoreline and subtidal areas and therefore, habitat restoration has been assumed in the development of this work plan.

Figure 7 diagrammatically illustrates a probable scenario for habitat restoration, coupled with cleanup of the riverbank soils. It has been assumed that most of the affected soil along the riverbank extends down to depths of 10 to 15 feet. The subtidal sediments below the riverbank are assumed to be affected by COCs in the upper 2 to 3 feet of sediment.

There are three habitat zones present on and adjacent to the shoreline:

- A degraded shoreline consisting of a steep armored bank that is dominated by invasive plants like the Himalayan blackberry that provide little or no habitat value;
- A shallow sloping intertidal bench composed of sand and mud; and
- A shallow subtidal zone between the intertidal bench and the navigation channel in the waterway.

The current shoreline provides little or no habitat value for fish and wildlife, nor does it enhance the value of adjacent habitats. The first two habitats are present within the property boundary, while the third subtidal habitat is adjacent to the site.

As shown in Figure 7, the probable scenario for habitat restoration is to excavate the soils and remove the rocks and concrete riprap from the shoreline. The base of the excavation would be sloped away from the barrier wall at a 2:1 slope. The excavation would continue to an approximate elevation of +5 feet above mean lower low water (MLLW). In these areas, suitable clean fill (such as "fish mix") would be placed, and new slopes would be restored at a flatter grade.

The upper 2 to 3 feet of sediment in the intertidal flats extending below +5 feet would also be excavated and removed out to an approximate elevation of +1 to +2 feet above MLLW. After this

material has been removed, suitable clean fill materials would be placed to restore the slopes to the original topographic contours.

The shoreline and the intertidal bench area would be planted with suitable native vegetation to provide habitat. Typical native species that could be planted in each area include:

- Shoreline-riparian habitat:
  - Black hawthorne (*Cartaegus douglasii*)
  - Shore pine (*Pinus contorta*)
  - Snowberry (*Symphoricarpos albus*)
  - Bald hip rose (*Rosa gymnocarpa*)
- Intertidal bench habitat:
  - Lyngby's sedge (*Carex lyngbei*)
  - Tufted hairgrass (*Deschampsia cespitosa*)
  - Softstem bulrush (*Scirpus validus*)
  - Arctic rush (*Juncus articus*)

On the southern edge of the site, the barrier wall is closer to the property line, which is approximately coincident with the current fence line. The Slip 6 shoreline is covered by a steep riprap-armored bank. The intertidal area in Slip 6 is very narrow and difficult to access except from a boat. Slip 6 is owned by the Boeing Company, and there are no foreseeable habitat restoration plans for Slip 6. Possible remedial options are limited and will likely include some type of sequestration or fixation if the COCs remaining in this area pose human health or environmental risks.

2.2 SAMPLING LOCATION AND FREQUENCY

Soil and groundwater samples will be collected during the shoreline investigation. Soil samples will be collected from the southern and western downgradient sides of the barrier wall. Groundwater samples will be collected from the southwest corner and Slip 6 shoreline areas. No groundwater samples are proposed to be collected from the Duwamish riverbank area. Groundwater monitoring in the area north of groundwater monitoring well DM-8 conducted since the installation of the barrier wall shows that concentrations of COCs in groundwater have decreased substantially. For this reason, no groundwater samples are proposed for the Duwamish riverbank sample locations.

The data quality objectives for the shoreline investigation are to provide data that allows:

- Determining the nature of COCs in the shoreline area that are anticipated to be removed during the habitat restoration;
- Determining the geotechnical properties of the shallow soils adjacent to the barrier wall to determine slope stability in the event of excavation;
- Determining the groundwater quality on the downgradient side of the barrier wall, especially in those areas with elevated concentrations of dissolved copper and elevated pH readings.

2.2.1 Southwest Corner and Slip 6 Shoreline Soil Samples

Geoprobe investigations conducted in 2000 and 2001 documented the presence of elevated concentrations of arsenic, copper, and mercury in groundwater samples collected next to Slip 6 and along the southernmost portion of the Duwamish shoreline. This area of Slip 6 and the southernmost portion of the Duwamish shoreline are referred to as the southwest corner of the site. These samples also showed that an area of elevated pH was present from the west well cluster of DM-8/MW-42 south along the wall and then east past the south well cluster of MW-43/MW-44. No soil samples were collected at the same time as these Geoprobe groundwater samples. Therefore, additional soil sampling is needed in these areas.

As shown on Figure 5, nine soil sample locations are proposed at the southwest corner. Table 3 summarizes the sample locations, sample depths, sample matrices, analytes, and analytical methods proposed to be used for these samples. Each boring will be completed using direct-push drilling to the approximate depth listed on Table 3 or until refusal. Each boring will be logged continuously. Six of the boring locations will be advanced to approximately 37 feet in depth, and three of the locations will be extended 20 more feet to approximately 57 feet in depth (the exact depth of the sampling intervals will depend on sample recovery and the specific tooling available on the direct-push rig). Soil samples will be collected in the upper 2 feet of the boring and then at approximate intervals of every 5 feet. Each soil sample will be analyzed for the entire suite of chemical analytes. The shallower borings will extend to the approximate depth of the performance monitoring wells screened in the upper portion of the shallow aquifer, while the three deeper borings will provide information about the current soil conditions at the depth of the performance monitoring wells screened in the lower portion of the shallow aquifer. The locations of these borings were selected to provide additional information for areas between the existing exterior performance monitoring wells as shown on Figure 5.

In addition, samples from the southwest corner will be analyzed for the geotechnical properties listed in Table 3; the specific samples selected for geotechnical analyses will be determined in the field. Geotechnical samples will be selected for analysis based on the general grain size and location of the proposed sample relative to the depth of the excavation to determine the maximum slope of

excavation that the barrier wall and soils can support. Samples from 10 and 15 feet in depth will be collected for geotechnical analyses from the three boring locations shown in Figure 5. The 15-foot depth is approximately equivalent to the 5-foot MLLW elevation envisioned as the base of the excavation. The geotechnical data collected during this investigation will be combined with the existing geotechnical data that was collected prior to construction of the barrier wall. This combined dataset should be sufficient for design requirements of the CMS.

As specified in the 2006 soil sampling QAPP (Geomatrix, 2006f), field duplicate samples will be collected at a rate of 10 percent or one duplicate for every 10 samples. A field equipment rinsate blank will be collected at a rate of 5 percent or once every 20 samples. The field equipment rinsate blank samples will be prepared by pouring analyte-free water though the decontaminated soil sampling equipment prior to beginning sampling activities at one of the nine sampling locations. A qualitative trip blank sample consisting of VOC-free water will be shipped with every cooler containing soil samples for VOC analysis (Geomatrix, 2006f).

2.2.2 Duwamish Riverbank Soil Samples

As shown on Figure 5, six soil sample locations are proposed along the riverbank of the Duwamish Waterway north of wells DM-8/MW-42 and south of MW-38R/MW-39. Soil borings will be installed, and sampling will be conducted in a manner similar to that for the southwest corner and Slip 6 samples, except the borings will be completed to an approximate depth of 15 feet. Samples for analysis will be collected in the upper 2 feet of the boring and then at intervals of every 5 feet to the total depth of approximately 15 feet or until refusal.

The depth of the borings was selected so that the borings would provide representative information about past impacts of site operations in soils along the shoreline. The soils along the shoreline will likely be excavated during habitat restoration, and the sampling results will identify disposal options available for these soils. The riprap and other debris in this area will likely be removed during restoration, but currently this material may prevent or hinder access for collection of samples from this area. If additional data related to the nature and extent of soil in the shoreline area are needed, the data will be collected during habitat restoration, prior to completion of the initial excavation.

Table 3 summarizes the proposed Duwamish riverbank sample locations, sample matrices, samples depths, analytes, and analytical methods. Testing will include both chemical and geotechnical analyses; the specific samples for geotechnical analyses will be collected at the three locations shown in Figure 5. Geotechnical samples will be selected for analysis based on the general grain size and location of the proposed sample relative to the depth of the excavation to determine the maximum slope of excavation that the barrier wall and soils can support. Samples from 10 and 15 feet in depth will be collected for geotechnical analyses. The 15-foot depth is approximately equivalent to the

5-foot MLLW elevation envisioned as the base of the excavation. The geotechnical data collected during this investigation will be combined with the existing geotechnical data that was collected prior to construction of the barrier wall. This combined dataset should be sufficient for design requirements of the CMS.

Field duplicate samples, equipment rinsate blank samples, and trip blanks will be collected in coordination with the southwest corner and Slip 6 samples, as described in Section 2.2.1.

2.2.3 Southwest Corner and Slip 6 Shoreline Groundwater Sampling

As mentioned previously, the 2000 and 2001 Geoprobe investigations documented elevated concentrations of several COCs in groundwater along Slip 6 and the southernmost portion of the Duwamish shoreline of the property. Ongoing performance monitoring of groundwater indicates that elevated pH and/or elevated concentrations of copper and toluene are still present in the southwest corner and along Slip 6.

In 2000 and 2001, a Geoprobe investigation was conducted by AGI along the shoreline area to evaluate concentrations of metals and pH in groundwater next to Slip 6 and the Duwamish Waterway. Although the information from this investigation is not extensive, the soil and groundwater sampling results did indicate high concentrations of copper and elevated pH in the southwest corner of the site and near the current location of MW-44 (AGI, 2001). It should be noted that these samples were collected prior to installation of the barrier wall in 2003, and concentrations of metals and pH may have changed in response to re-equilibration of groundwater flow paths on the downgradient side of the barrier wall.

During construction, two sections of the barrier wall needed to be re-installed, one near the southwest corner and a second along the south end (Slip 6 area). In the southwest corner, large submerged tree trunks were encountered at depths of 13 to 30 feet during wall installation. After the vibrating beam construction equipment became stuck, these trunks were excavated using a large hydraulic excavator and then removed. Once the tree trunks were removed, this area of the barrier wall was subsequently re-installed by replacing barrier wall panels in the area of the tree trunks. A smaller unidentified obstruction (4 feet wide) was also encountered along the south side (Slip 6) of the barrier wall during installation. The obstruction was overcome by drilling through it, and the barrier wall in this area was also addressed with re-installation of barrier wall panels (RCI, 2003). The locations of these two reinstalled areas are shown on Figure 5.

Additional groundwater sampling is proposed in the southwest corner and along the Slip 6 shoreline. Figure 5 shows the location of the proposed groundwater sampling locations. Table 3 summarizes the proposed groundwater sampling locations, the sample depths, analytes, and analytical methods.

The groundwater samples will be collected adjacent to each of the nine soil sample locations using a dedicated, direct-push groundwater boring. At the six shallower soil sample locations, five groundwater samples will be collected at approximate 5-foot intervals to a maximum depth of 35 feet, which is equivalent to the screen interval for the Upper Zone monitoring wells at the site. At three of the locations (as shown in Figure 5), four additional groundwater samples will collected up to a depth of 55 feet (the exact depth of the groundwater sampling intervals will depend on sample recovery and the specific tooling available on the direct-push rig). These deeper groundwater samples will allow further resolution of groundwater quality in this area of elevated pH readings and copper concentrations in groundwater samples.

In addition to the analyses requested by EPA, between four and six samples of water will be selected for analysis of general water chemistry. These samples will be collected on the basis of field pH measurements and spatial variability and analyzed for major cations, major anions, alkalinity, density, and total dissolved solids as shown in Table 3. At least two of these samples will be collected from deeper portions of the shallow aquifer from the three borings that extend to 55 feet.

The groundwater sampling and analytical methods were selected to identify the geochemical conditions present within the thin strip of sediment between the barrier wall and Slip 6 and to allow the selection of appropriate remedial approaches that could be applied in this area.

As specified in the 2002 groundwater monitoring QAPP (URS, 2002b), field duplicate samples will be collected at a rate of 10 percent or one duplicate for every 10 samples. A field equipment rinsate blank will be collect at a rate of 5 percent or once every 20 samples. The field equipment rinsate blank samples will be prepared by pouring analyte-free water though the decontaminated groundwater sampling equipment prior to beginning sampling activities at one of the nine sample locations. A qualitative trip blank sample consisting of VOC-free water will be shipped with every cooler containing groundwater samples for VOC analysis (Geomatrix, 2006g).

2.2.4 Pore Water Samples

Two pore water samples will be collected from the north side of Slip 6, from the narrow intertidal zone at the base of the riprap that armors this shore; Figure 8 shows the location of historical pore water sampling locations. Pore water samples were collected from seeps located on and near the site in 1991 (Landau, 1991); during Round 3 of the RFI in 1995 (CH2M HILL, 1995); and in 2004 using specialized pore water sampling techniques (EPA, 2005). Prior to the EPA investigation, the seep water samples were collected by immersing bottles into the seeps. This method of sample collection may have biased the analytical results due to the potential introduction of fines and sediment. During the EPA investigation, small-diameter probes (MHE PushPoint samplers) were installed as temporary

piezometers in the intertidal areas. Pore water samples were collected from these probes using portable peristaltic pumps (EPA, 2005).

AMEC proposes to collect two pore water samples from the intertidal slope along the north side of Slip 6 (Figure 9). One sample will be located in the intertidal area shoreward of the SHB-5 sampling location. The other pore water sample will be collected from the intertidal area shoreward of SBH-18. These two locations are close to the exterior performance monitoring well clusters that still have elevated copper concentrations and pH readings as discussed in Section 1.1. The analytical methods and quality assurance/quality control (QA/QC) methods for pore water samples are similar to those for groundwater samples; therefore, pore water sampling is included in this section of the work plan. Due to the logistical and safety concerns, this sampling will likely be conducted during the sediment investigation.

2.3 SAMPLE DESIGNATION

Each sample collected will be given a unique sample identification number. Sample identification numbers will include the site name (former Rhone-Poulenc or RP), the sample date (mmddyy), and a sample sequence number. For example, a sample ID of RP052311-03 would identify the third sample collected on 05/23/2011. The sampling sequence number will not include the boring number or indicators of field blanks, equipment blanks, etc. A master sampling log that documents the sequence numbers and the corresponding wells will be maintained by field personnel in the field logbook.

2.4 SAMPLING EQUIPMENT AND PROCEDURES

This section describes the equipment and procedures to be used during sampling events.

2.4.1 Soil Sampling

Table 4 lists the sample containers required for soil sampling. All soil samples will be collected using direct-push drilling equipment with a 2-inch outside diameter sample rod—these rods are typically 4- to 5-feet in length. The soil samples will be collected using acetate liners to minimize sample loss. All borings will be logged continuously by an AMEC geologist. Although the borings will be logged continuously, the volume of sample recovery may vary for a given interval, and the recovery will be noted by the field geologist. Lithology will be described by the field geologist using the Unified Soil Classification System (USCS) (ASTM, 2009).

Once the core has been described, the field geologist will collect soil samples. Samples will be placed into the appropriate precleaned and labeled sample container using decontaminated stainless steel spoons. The sampler will wear a fresh pair of disposable nitrile gloves to collect the samples. All VOC soil samples will be collected following EPA Method 5035 soil sampling procedures, and the VOC soil sample aliquots will be stored in 40-milliliter (mL) glass vials. Real-time photoionization

detector (PID) readings will be collected from small aliquots of soil placed into polyethylene bags to screen for the presence of VOCs. All prelabeled bottles will be sealed, the outside of the bottle will be cleaned of loose soil using disposable paper towels, and the bottles will placed into a cooler.

All sampling equipment (drill rods and spoons) will be decontaminated using either a hot-water pressure washer (typically used for decontamination of drill rods) or a three-step process consisting of washing in water containing Alconox, a rinse in clean tap water, and a final rinse with deionized water using spray bottles or brushes. Decontamination water will be collected in buckets with secondary containment using polyethylene mortar tubs to catch spillage.

2.4.2 Groundwater Sampling

Table 5 lists the sample containers required for groundwater sampling. The groundwater samples will be collected using a Hydropunch direct-push groundwater sampler (or equivalent). The sampler will be driven into the soil within a radius of approximately 1 foot from the associated soil sample locations. The sampler will be advanced to the initial planned sampling depth shown in Table 3. The drive rod will be retracted approximately 4 feet to expose the screen at the end of the Hydropunch sampler. Disposable polyethylene tubing will be lowered into the screen section, and a peristaltic pump will used to extract the groundwater sample from the Hydropunch. Groundwater samples will be collected following low-flow sampling techniques using a flow-through cell. Stabilization parameters, consisting of temperature, specific conductivity, pH, and oxidation/reduction potential (ORP), will be recorded to determine when purging is complete; due to the temporary nature of the groundwater sampling method, turbidity readings will not be used to determine when purging is complete. The low-flow sampling procedures and criteria outlined in the 2002 groundwater sampling QAPP (URS, 2002b) will be followed, with the exception of the use of a peristaltic pump and the need to stabilize turbidity prior to collecting a sample. All stabilization readings will be recorded on standard AMEC low-flow groundwater data sheets.

The sample tubing will be disconnected from the flow-through cell and the groundwater discharge will be directed into a 5-gallon polyethylene bucket between samples. Once the sample is collected, the bottle will be sealed; the samplers will check all samples for analysis of VOCs to verify that the samples are free of bubbles. The sampler will check Table 5 to confirm that all groundwater samples have been collected for the specified analyses and in the appropriate prelabeled bottles. The exterior of bottles will be wiped down with a clean paper towel before being stored on water ice in a cooler.

All sampling equipment (especially the Hydropunch sampler) will be decontaminated after collection of each sample. Decontamination will be performed as described in Section 2.4.1 for soil sampling.

Once decontaminated the Hydropunch sampler will be driven back down the same borehole to the next deeper 5-foot sampling interval and another groundwater sample will be collected following the procedure described above. The process will be repeated until the target depth is reached.

2.4.3 Pore Water Sampling

Two pore water sample locations along the Slip 6 intertidal area will be sampled using PushPoint samplers. The detailed PushPoint sampling methodology is described in Appendix B. With this methodology, a 6-foot-long stainless steel MHE sampler is pushed into the intertidal sediments to a depth of approximately 3 feet. A small-diameter polyethylene tube is connected to the sampler using a shorter connecting length of flexible tubing. A peristaltic pump will be used to purge water from the PushPoint sampler at a rate of 50 to 200 milliliters per minute (mL/minute). During the purge, the water will be pumped through a flow-cell equipped with a multiparameter probe (such as a Horiba U-22) to record changes in general parameter readings (temperature, electrical conductivity, pH, ORP, and dissolved oxygen). When the general parameter readings stabilize and the purged water is free of visible sediment, a pore-water sample will be collected.

2.5 Sample Handling and Analysis

Tables 4 and 5 summarize the sample bottles, preservation methods, and holding times for soil and groundwater, respectively. Figure 10shows a copy of a typical chain of custody form that will be used to submit soil or water samples.

The sampler(s) will record all sample numbers in the field logbook using "Rite-in-the-Rain" pens or equivalent, creating a record of which samples were collected at which locations, and noting the sampling depths and analytes. This information will be cross-checked with the information provided in the chain of custody form to verify that both are accurate. The field logbook will be used to document activities, weather conditions, and visitors to the site, and any departures from procedures during the investigation. Any mistakes in the field notes or chain of custody form will be crossed out with a single line and annotated and initialed by the person making the correction.

All samples will be transported in a cooler to the analytical laboratory by a field vehicle or laboratory vehicle. None of the samples are anticipated to be shipped via air freight. When custody of the samples is changed, the sampler and the new sample custodian will sign and date the chain of custody form. Field documentation may also include digital photographs of the sampling equipment, soil samples, field activities, or any other relevant subject material.

All acetate liners used for soil sampling, gloves, paper towels, and other debris will be placed in a trash bag for disposal as refuse. All soil cuttings will be consolidated into 55-gallon open-top drums to be located at the HCIM groundwater pretreatment building (the location of the pretreatment building is

shown on Figure 2). The drums will be sealed, labeled, and disposed of in accordance with all state and federal requirements. The borings that are the source of the cuttings in each drum will be recorded in the field logbook. If the total metals analytical results of one of the borings in a given drum exceeds 20 times the toxicity characteristic leaching procedure (TCLP) regulatory limits for arsenic, barium, cadmium, chromium, lead, mercury, selenium, or silver, then a portion of the sample will be analyzed using the TCLP. If the results of this analysis are below the TCLP regulatory limits for the respective metals, then the drum will be disposed of as nonhazardous waste. If the samples from the borings contributing to the drum contained detectable concentrations of toluene, then the drum will be disposed of as U220 waste. This approach is consistent with the approach specified in the *Affected Soil Removal Plan* (Geomatrix, 2006g), which was approved of by the EPA and followed during redevelopment of the West Parcel.

All peristaltic polyethylene tubing used for collecting groundwater samples will be disposed of between sampling points. The collected purge water will be discharged to the King County POTW following pretreatment using the HCIM pretreatment system pump/sump tank, following procedures outlined in the revised *Operation, Monitoring, Inspection, and Maintenance Plan* (OMIMP) (AMEC, 2009). All tubing, gloves, and paper towels will be bagged and disposed of as refuse.

All work conducted during the shoreline investigation will be conducted in accordance with the 2006 soil sampling QAPP (Geomatrix, 2006f) or the groundwater monitoring QAPP (URS, 2002b). Since these QAPPs were drafted, there have been slight changes in analytical methods or new analyses that were not covered under the original QAPPs. Table 6 presents a list of the differences between the existing QAPPs and the methods proposed in Tables 4 and 5, and summarizes how these differences will be resolved in the shoreline investigation. Copies of the standard operating procedures (SOPs) for the new analyses and sampling methods are presented in Appendix B.

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3.0 SEDIMENT SAMPLING AND ANALYSIS PLAN

Previous sediment sampling conducted in the vicinity of the project site is summarized in Section 1.1 and in Table 1. Previous surface and subsurface sample locations are shown on Figure 8. Sampling locations with results that exceed the SMS criteria are also shown on Figure 8.

Sediment samples will be collected in the offshore area within and adjacent to the IAAI Lease Property to assess the nature and extent of the Sediment Management Standards (WAC 173-204) COCs. In addition, sediment samples will be analyzed for dieldrin and vanadium. Sediment samples will include both grab and core samples of sediments. This section describes the objectives of the sediment investigation and the sampling methods and procedures that will be used. All of the sediment sampling and analysis work will be conducted following the guidelines and protocols described in the project-specific QAPP prepared for the sediments investigation and presented in Appendix A.

3.1 SAMPLING OBJECTIVES

The intent of the core and grab sampling is to collect sediment samples for analytical testing to assess the nature and extent of potential surface and subsurface contamination in the tideflat and offshore areas that could be attributed to potential sources on the upland portions of the site. The results of this sediment investigation and the previous sampling efforts will be reviewed for trends in COC concentrations that indicate whether the source(s) of contamination can be attributed to offshore transport of onshore sources of contamination or whether historical releases from the site may be responsible for the observed trends in COC distribution. Core sampling is also used to determine the depth of contamination and the presence of COCs in the native alluvial sediments. The contact with native sediments is expected to be characterized by a sharp transition between the relatively unconsolidated, recently deposited sediments and the more compacted, poorly sorted, and sandy native material. Vertical and lateral changes in COC concentration will be coupled with the physical nature of the sediments to determine the possible sources of the observed distribution of COCs in the sediments. Table 8 presents the sediment action levels and the associated method detection and reporting limits.

EPA has identified the COCs for the sediments at the project site as the constituents specified in the Washington State Marine SQS (WAC 173-204-320), as well as dieldrin and vanadium. Total organic carbon (TOC) will be analyzed for each sample to allow organic carbon normalization of organic analytes for comparison to the SQS chemical criteria. Total solids will be determined for all samples. Grain size will be determined for surficial sediments collected using the grab sampler. Grain size will not be determined for subsurface sediments because of the limited sample volume available.

A majority of the proposed sample locations were laid out in a systematic, triangular grid in the tideflat and offshore areas adjacent to the uplands portion of the site (Figure 9). In response to comments from EPA, additional judgmental samples will be included in the sediment investigation. Additional grab and core samples will be collected to investigate sediment quality near a groundwater seep off the southwest corner of the site (near the mouth of Slip 6). A surface sample will also be collected as close as possible to a previous intertidal sample location with elevated mercury results on the north bank of Slip 6 (location 04-intsed-3; Figure 8). Historically several outfalls from the former industrial site discharged into the Duwamish, and Figure 6 shows the location of these historic outfalls. The systematic grid being used results in samples being collected in the vicinity of all outfalls discharging to the Duwamish. The King County outfall that historically and currently discharges to Slip 6 was evaluated in the EPAs sediments investigation. Further sediment sampling is not warranted in Slip 6 beyond the locations proposed due to fact that Slip 6 based on EPA sampling, appears to have been dredged.

3.2 SAMPLING LOCATION AND FREQUENCY

Cores and grab samples will be collected at 20 sample locations (17 on the triangular grid and 3 additional judgmental sample locations: one at the southwest corner of the IAAI Lease Property and two on the north side of Slip 6 [Figure 9]). A differential global positioning system (GPS) unit with submeter precision will be used to navigate to the sample locations. Proposed and actual sample locations will vary slightly due to vessel positioning, tidal or river currents, and winds. Similarly the core and grab sample locations will be located at approximately the same location but could vary due to the factors noted above. The GPS will be preprogrammed with the sample locations prior to the start of field activities. At the start and end of each field day, the GPS receiver antenna will be positioned near a known location or check point to confirm that the unit is still providing accurate readings. The check point data will be logged at the beginning and end of each day. Navigation data will be logged electronically into the GPS at each sampling station and written on the sample collection form. The actual sample location (core or grab) will be logged using the on-board GPS.

3.3 SAMPLE DESIGNATION

All samples will be assigned a unique alphanumeric identification code. The code for grab samples will consist of the site name (former Rhone-Poulenc or RP) and a sequential location code (e.g., RP-01). For the core samples, the identification code will also include a segment or sample-depth identifier (e.g., RP-01-0020). The sequential location codes during this investigation will start with 01 and continue through 20. Locations where duplicate grab or core samples are collected will be assigned sequential location codes (i.e., RP-20, RP-21) that do not identify the paired location.

3.4 SAMPLING EQUIPMENT AND PROCEDURES

Surficial sediment samples collected in the tideflat and offshore areas will be collected using a grab sampler to collect the top 0.33 foot (10 cm) of sediment. An impact corer will be used to collect subsurface cores from the surface of the sediment to a maximum depth of 15 feet below the mudline. The cores will then be prepared, and samples will be collected from the sample tube at intervals corresponding to 1-foot in situ depth intervals.

Sediments will be collected adjacent to previous intertidal sample location 04-intsed-3 on the north side of Slip 6 using hand collection methods (stainless steel spoons or trowels). The location is on a steep partially-armored slope, and the use of grab samplers or coring devices is impractical.

3.4.1 Core Samples

Subsurface sediment cores will be collected using an impact corer. The impact corer uses the impact from a linear pneumatic hammer, delivering approximately 300 blows per minute to drive a 4-inch-square aluminum core into the sediment. This allows for a continuous core sample to be collected over the depth that the tube is driven. The bottom of each core tube will be fitted with a hinged core catcher to prevent loss of the sediment during extraction. Core tubes are single-use and will be decontaminated prior to arrival at the site in accordance with Section 3.4.4. Core sample locations will be located with the on-board GPS. Sample locations will be recorded for each attempted core. Water depth at each core location will be determined using a weighted lead line.

Table 7 lists the proposed sample locations, including Washington state plane coordinates. Table 9 specifies the core depth intervals to be sampled, provides a list of the collected samples that are proposed to be analyzed initially, and lists the proposed initial analyses to be performed for each sample location. Field duplicate core samples will be collected at two sampling locations within 6 feet (approximately 2 meters) of the original core location.

Paired penetration and recovery measurements will be used to account for thinning and compaction of the sediments during driving. An on-deck top-of-sediment measurement from the top of the core tube to the surface of the sediment within the core tube will be made to account for any movement or loss of sediment in the core tube as the core is withdrawn. The penetration and recovery data and the on-deck top-of-sediment measurement will be entered into a spreadsheet program to generate a boring log.

3.4.1.1 Core Acceptability Criteria

Full core penetration and sample recovery may not be possible at all locations using the proposed sampling equipment. Native sediments can be very dense, and recovery of deeper sample intervals

becomes difficult with greater penetration. If penetration is less than the proposed target depth, then cores will be evaluated to determine their acceptability using the following protocol:

- If the contact with native sediments is present in the core and samples can be collected below the contact, and the core was driven to refusal using the available equipment, additional sampling attempts at that location may not be required.
- If penetration is less than the target depth and the native contact is not visible in the core or if samples cannot be collected below the contact, the corer will be relocated a minimum of 6 feet (2 meters) from the original location and a second core will be attempted.

If a second core is attempted, penetration is less than the target depth, and no native contact is visible or samples cannot be collected below the contact, no further sampling using the impact corer will be attempted at that location. If deeper sediment samples are needed at a location to characterize the sediments, then an additional round of sampling using other equipment may be required.

3.4.1.2 Core Processing

At all core sampling locations, discrete samples will be collected from each core from the 1-foot in situ depth intervals to the target core depth of 15 feet below mudline or until refusal.

If the volume of recovered sediment available within a depth interval is insufficient to perform all the analyses identified in Table 9, additional sediment volume from the next deeper interval will be added to provide sufficient sample volume. The next subsequent sample will be collected from the next complete, intact 1-foot in situ depth interval. This sampling routine may be modified in the field based on site conditions at the direction of the field geologist.

Core processing will be conducted following the health and safety requirements specified in a Site-Specific Health and Safety Plan (to be prepared to address core processing and work to be performed on-board vessels on the water). The handling and processing of sediment cores will occur within a secured exclusion zone using Level D personal protective equipment (PPE). Only one core tube will be handled and processed at a time. Cores will be held for a maximum of 24 hours before processing. Unprocessed cores held more than 8 hours will be chilled with ice. Core tubes will be transported and stored horizontally.

Procedures will be followed during processing of the cores to minimize the effects of carry-down of shallower and potentially more contaminated sediments into deeper, less contaminated sediments. Carry-down may result from wall friction between the sediment and the inside surface of the core tube. This form of carry-down is evident as a bending or a downward deflection of a horizontal soil stratum near the edges of the core tube. Carry-down may also result from sediment with low cohesive properties collecting behind the hinged core catcher and being carried down one side of the

core tube. Carry-down may contaminate clean, deeper strata with contaminated sediments from shallower strata, confusing the interpreted distribution of chemical contamination within a core.

The effects of carry-down while processing the collected cores will be minimized by using the following steps:

- The core tube will be placed on sawhorses and oriented with the hinged side of the core catcher to the side.
- The uppermost side of the core tube will be removed using a circular saw. The depth of cut on the saw will be set to just slightly greater than the wall thickness of the aluminum tube.
- The core will be inspected for significant loss of sediment from the bottom of the core tube and for separation (gaps) along the length of the recovered sediment.
- If the core is acceptable, a thin layer (approximately 1-centimeter or 0.38-inch thick) will be removed from the exposed surface of the sediment with a decontaminated stainless steel scraper.

The surface layer of sediment will be removed starting at the bottom of the core tube and moving toward the top. This method minimizes potential contamination of clean, deeper layers with material from shallower, potentially more contaminated layers.

The exposed surface of the sediment core will be photo-documented using either photographs or video. A qualified field geologist will log each core using USCS classifications (ASTM, 2009) and note the presence of any soil structures, odors, or visible oil sheens. Sediment descriptions and the interpreted in situ depths of each sediment horizon will be transcribed into a summary log.

Stainless steel plates will be inserted between each 1-foot in situ depth interval to minimize carry-down and prevent cross-contamination between depth intervals. Sediment samples will be collected at the center of the core from each 1-foot sampling interval starting from below each inserted plate and extending down the core tube until the next inserted plate. Equal amounts will be taken throughout the interval to ensure that the sample material is representative of the entire depth interval. If there is insufficient material available within the 1-foot sampling interval, the interval may be extending farther down the tube (deeper) until sufficient sample volume is obtained. Approximately 1 liter of sediment will be needed for all of the analyses listed in Table 9. The distance down the tube that sediment is removed will be recorded to provide information on the actual depth interval where each sample was collected.

Sediment from each sampling interval will be placed directly from the core tube into 1-liter glass jars. Sediment will not be homogenized before being placed in the jar. Equal amounts of sediment from

the entire segment length will be placed in the sample jar so that the sample will be representative of the entire segment. . Additional sediment remaining within a sample interval after collection of the required 1 liter of sediment may be collected for geotechnical testing (e.g., bulk density, percent solids, Atterberg Limits, percent moisture content, specific gravity, etc.) at the direction of the field geologist. Samples for geotechnical testing will not be frozen if freezing could alter the physical properties being tested.

Each sample container will be labeled with a preprinted sample label. The sample label will contain the project number, sample identification, analyses, date and time of collection, and initials of the person(s) preparing the sample. The label will also state that the sediment is not homogenized. The sample containers will be placed in a cooler with "blue ice" for transport to the laboratory. The list of core samples (representing core segments or intervals) proposed to be analyzed is presented in Table 9. Analysis of samples representing the 2- to 3-foot, the 4- to 5-foot, the 8- to 9-foot, and the 12- to 13-foot intervals will provide information on the depth of sediments that exceed the SMS criteria. Samples from core segments not analyzed initially will be archived at the project laboratory for possible future analyses. Additional analyses may be undertaken on archived samples to refine the depth of contamination using an iterative decision process.

Mercury has a 28-day holding time for frozen sediments. Mercury analysis in archived sediments may exceed the holding time, and the data will need to be qualified if reported. Mercury in sediments in the vicinity of the project site does not appear to be present at concentrations above the SQS except at two locations. The results from the core samples analyzed within the holding time will be used to determine the vertical distribution of mercury at the project site.

A chain of custody form will be filled out for the samples, placed in a resealable plastic bag, and placed in the cooler with the samples (Figure 11). The chain of custody form will state that the sample is not homogenized and that the entire sample volume must be fully homogenized in the laboratory before being analyzed.

3.4.2 Grab Sample Collection

Surficial grab samples will be collected using a modified stainless steel, 0.2-square-meter pneumatically operated grab sampler deployed from the sampling vessel. The sampler will be decontaminated prior to arrival at the site in accordance with Section 3.4.4. The planned sampling locations are shown on Figure 9. Grab sample locations will be located in the field using the on-board GPS at approximately the same location as the corresponding core sample location given the constraints of vessel positioning, tidal and river currents, and wind. Sample locations will be recorded for each attempted grab. Water depth at the grab location will be determined using a weighted lead line.

The sampler will be deployed and retrieved with minimum swinging while out of the water. Excessive swinging can cause the sampler to trigger prematurely upon deployment and disturb the sediment sample upon retrieval. Swinging will be minimized by heading the survey vessel into any waves when the sampler is out of the water and by attaching handling lines to the cable operated by the sampling team.

When the device is lowered into the water too quickly a bow wave can form at the front of the sampler that could disturb the sediment; therefore, it is essential that the sampler enter the sediment at a relatively slow speed. The lowering speed of the sampler upon entering the sediment must be approximately 1 foot per second (0.3 meter per second) or less. Lowering rates through the water column can be faster until the sampler is several meters from the bottom as long as the speed at sediment entry is 1 foot per second or less. Swell and chop can significantly degrade sample quality because of effects on the entry speed of the sampler (vertical ship motion alternately adds to and subtracts from entry velocity). These factors will be considered when swell and chop are present.

After the sampler contacts the bottom, it will be initially retrieved slowly to permit the device to close properly. After the jaws are closed, a constant retrieval speed will be used to avoid jerking the sampler and possibly disrupting the sample. The sampler will be secured as soon as possible after being brought on board. Field-duplicate grab samples will be collected at two sampling stations within 6 feet (approximately 2 meters) of the original grab sample location.

Processing steps for grab samples include initial inspection and acceptance of a grab sample, photography, sediment description, and sediment collection and homogenization. All of the grab samples collected will be analyzed for the list of analytes presented in Table 9. Surplus sample volume will be archived at the project laboratory for possible future analyses.

3.4.2.1 Sample Acceptability Criteria

After the sampler has been secured, the sediment sample will be inspected carefully before being accepted. The sample will be inspected for acceptability based on adherence to the following acceptability criteria:

- The sampler is not overfilled with the sample such that the sediment surface is pressed against the top of the sampler.
- Overlying water is present (indicates minimal leakage).
- The overlying water is clear (excessive turbidity in the overlying water indicates that the sample has been disturbed).
- The upper sediment surface is relatively flat (indicates minimal disturbance or winnowing).

- The penetration depth is at least 0.5 foot for a 0.33-foot-deep (15 cm for a 10-cm-deep) surficial sample.

If a given sample does not meet any one of these criteria, it will be rejected. If the sample is acceptable, the overlying water will be removed. The water will be slowly siphoned from one side of the sampler with minimal disturbance to the sample.

3.4.2.2 Photography and Sediment Description

Once the overlying water has been removed, the surface of the grab sample will be photographed using a digital camera. The digital camera will be mounted on a removable bracket that attaches to the grab sampler to provide a consistent field of view.

A qualitative sample characteristics form is filled out for each acceptable grab sample documenting the surface and subsurface sediment characteristics, including penetration depth.

3.4.2.3 Sediment Collection and Homogenization

The top 0.33 foot (10 cm) of sediment will be collected with a stainless steel spoon from the center of the grab for analysis. Sediment touching the stainless steel sides or bottom of the sampler will not be collected for testing or analysis. The sediment will be thoroughly homogenized in a stainless steel bowl using a stainless steel spoon. The grab sampler will be rinsed free of any sediment between attempts.

Each sample container will be labeled with a preprinted sample label. The sample label will contain the project number, sample identification, analyses, date and time of collection, and initials of the person(s) preparing the sample. The sample containers will be placed in a cooler with "blue ice" for transport to the laboratory. The list of samples proposed to be analyzed initially is presented in Table 9. Samples not analyzed initially will be archived at the project laboratory as described in Section 3.4.5 for possible future analyses.

A chain of custody form will be filled out for the samples, placed in a resealable plastic bag, and placed in the cooler with the samples (Figure 11).

3.4.3 Hand Collection of Surficial Sediments

Surficial sediment samples may be collected from the intertidal area using stainless steel utensils (spoons or trowels) if required. Sediments will be collected within 6 feet of the proposed sample location and to a depth of 0.33 feet, if possible. The sample location will be located in the field using a handheld GPS. The sample location will be logged, and the sediment surface at the sample location will be photographed using a digital camera. The minimum sample volume is 2 ounces (oz); however,

up to 1 liter of sediment may be collected. The proposed initial sample analysis schedule is presented in Table 9. Sample volume not analyzed initially will be archived at the project laboratory for possible future analyses.

3.4.4 Equipment Decontamination

Sample containers, instruments, working surfaces, technician protective gear, and other items that may come into contact with sediment sample material must meet high standards of cleanliness. Sample containers will be provided by Analytical Resources, Inc., and will be precleaned, certified, and individually labeled with a lot number traceable to a Certificate of Analysis.

The AMEC standard decontamination procedure for the core tubes and the grab sampler and other sample handling equipment is modeled after Puget Sound Estuary Program (PSEP) protocols (PSEP, 1997); however, the decontamination procedure will not use any acid or solvent rinses (the final rinse will be done using distilled water).

All stainless steel bowls, core dividers, and spoons will be decontaminated prior to the start of fieldwork and wrapped with aluminum foil to prevent recontamination. If it is necessary to decontaminate this equipment in the field, decontamination of field sampling equipment will be performed using the following procedure:

1. Prewash rinse with tap water.
2. First wash with solution of tap water and Alconox soap (brush).
3. Second rinse with tap water.
4. Second wash with solution of tap water and Alconox soap (brush).
5. Final rinse with tap water.
6. Final rinse with distilled water.
7. Coverage (no contact) of all decontaminated items with aluminum foil.
8. Storage in clean, closed container prior to use.

The single-use core tubes and grab sampler will be precleaned prior to arrival at the site using the procedure described above. In addition, all equipment and instruments used to remove sediment from the core or the sampler or to homogenize samples will be stainless steel.

The grab sampler will be rinsed free of sediment between attempts using surface water. Sediment adhering to the sampler will not affect the sample since only sediment from the center of the grab sample will be collected for analysis.

3.4.5 Sample Handling and Analysis

Table 9 provides a list of the sediment samples proposed for analysis. Analyses will be performed on samples collected from the 0- to 0.33-foot surface layer and from selected 1-foot depth intervals (Table 9). The selected 1-foot depth intervals will be used to determine the concentrations of the SMS list of analytes at defined depths below the sediment surface. Additional samples collected and archived pending the results of the initial analysis round may be analyzed to refine the depth of contamination using an iterative decision process.

Field-duplicate samples will be analyzed at a frequency of approximately 10 percent. Additional core intervals may be analyzed during the initial analysis phase based on sediment characteristics observed by the field geologist during core processing. Analytical methods, data quality objectives, and QA/QC procedures are specified in the project-specific QAPP prepared for the sediments investigation and included in Appendix A. Surplus sediment sample volume will be frozen (-18 degrees Celsius [°C]) and archived at the analytical laboratory. All sediment samples collected but held pending analysis will be archived at the analytical laboratory as described in the project-specific QAPP (Appendix A). Depending on the results of the initial round of analyses, additional analyses may be conducted in consultation with EPA.

4.0 DELIVERABLES

The following deliverables will be prepared during the shoreline and sediments investigation:

- A Joint Aquatic Resource Permit Application (JARPA) will be prepared and submitted to the Washington Department of Fish and Wildlife (DFW) in order to obtain a Hydraulic Project Approval (HPA) for the sediment investigation activities. A copy of the submitted JARPA will also be provided to EPA.
- A Shoreline Investigation Report will be prepared and submitted to EPA.
- A Sediment Investigation Report will be prepared and submitted to EPA.

As noted in Section 5.0, it is probable that the shoreline and sediment investigations will be conducted separately.

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5.0 SCHEDULE

This section presents the proposed schedule for conducting the shoreline and sediments investigations. Because of the need for additional permitting for the sediment investigation and the need to schedule the sediment investigation for a specific portion of the tidal cycle, it is probable that the shoreline and sediment investigations will be conducted independently. The results of the shoreline and sediment investigations may be reported independently or as a combined report, depending on the timing of the investigations.

5.1 SHORELINE INVESTIGATION

The schedule for the shoreline investigation will depend on the time frame for EPA's approval of the work plan and on scheduling availability of a qualified driller. Field activities are expected to take up to 2 weeks to complete; it is likely that laboratory testing will take another 3 to 4 weeks. A draft report will be submitted to EPA within 60 days of receipt of the last analytical results from the laboratory or within 180 days of receipt of approval of this work plan, whichever is earlier.

5.2 SEDIMENT INVESTIGATION

The schedule for the sediments investigation depends on the time frame for EPA to approve this work plan and on the timing of receipt of the HPA from the Washington Department of Fish and Wildlife. Field work will start within 4 weeks following EPA approval of the work plan and issuance of the HPA, whichever occurs later. Field activities are expected to take 4 to 5 days to complete. After the initial analytical results have been received from the laboratory, a draft report will be submitted to EPA within 60 days of receipt of the last batch of data from the analytical laboratory or within 180 days of receipt of approval of this work plan, whichever is earlier.

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6.0 HEALTH AND SAFETY

Worker health and safety requirements will follow a Site-Specific Health and Safety Plan prepared in accordance with applicable state regulations for hazardous waste site workers (WAC 296-843). Shoreline work will be conducted following the procedures specified in the existing health and safety plan for the site that was developed and used during previous soil and groundwater sampling events. A new health and safety plan will be developed to address sediment core processing work as well as work done on the water during sediment grab and core sampling.

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TABLES

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TABLE 1

SUMMARY OF HISTORIC SEDIMENT ANALYTICAL DATA [1]

Former Rhone-Poulenc Site
Tukwila, Washington

Chemical Parameter	Number of samples with non-detects	Number of samples with detects	Total number of sample results	Detection frequency (%)	Maximum detected value	Number of results exceeding SQS [2]	Number of results exceeding CSL [2]
Metals (ppm)							
Arsenic	0	104	104	100%	26.2	–	–
Cadmium	41	48	89	54%	1	–	–
Chromium	0	106	106	100%	41.1	–	–
Copper	0	106	106	100%	175	–	–
Lead	0	104	104	100%	133	–	–
Mercury	11	93	104	89%	1.8	2	2
Nickel	0	88	88	100%	33	–	–
Silver	39	23	62	37%	1	–	–
Zinc	0	106	106	100%	212	–	–
SVOCs (ppb)							
Total LPAHs	11	76	87	87%	7,100	–	–
Naphthalene	46	10	56	18%	440	–	–
Acenaphthylene	48	8	56	14%	20	–	–
Acenaphthene	67	20	87	23%	1200	2	–
Fluorene	56	31	87	36%	1000	1	–
Phenanthrene	11	76	87	87%	3,900	1	–
Anthracene	25	62	87	71%	500	–	–
2-Methylnaphthalene	46	10	56	18%	260	–	–
Total HPAHs	3	84	87	97%	20,000	1	–
Fluoranthene	6	81	87	93%	5,300	2	–
Pyrene	6	81	87	93%	4,100	–	–
Benzo(a)anthracene	11	76	87	87%	1400	–	–
Chrysene	8	79	87	91%	2100	–	–
Benzo(b)fluoranthene	3	72	75	96%	2000	–	–
Benzo(k)fluoranthene	10	65	75	87%	1700	–	–
Total benzofluoranthenes	3	78	81	96%	3700	–	–
Benzo(a)pyrene	12	75	87	86%	1400	–	–
Indeno(1,2,3-cd)pyrene	6	81	87	93%	1200	3	–
Dibenzo(a,h)anthracene	20	67	87	77%	700	23	1
Benzo(g,h,i)perylene	15	72	87	83%	1100	2	–
Chlorinated Benzenes (ppb)							
1,2,4-Trichlorobenzene	50	6	56	11%	3.6	–	–
1,2-Dichlorobenzene	52	4	56	7%	1.8	–	–
1,4-Dichlorobenzene	52	4	56	7%	2.3	–	–
Hexachlorobenzene	50	6	56	11%	1.2	–	–
Phthalates (ppb)							
Bis(2-ethylhexyl) phthalate	25	62	87	71%	2100	1	–
Butyl benzyl phthalate	12	44	56	79%	95	–	–
Diethyl phthalate	84	3	87	3%	2,700	1	1
Dimethyl phthalate	33	23	56	41%	83	–	–
Di-n-butyl phthalate	45	11	56	20%	35	–	–
Di-n-octyl phthalate	76	11	87	13%	2,000	1	–

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Former Rhone-Poulenc Site
Tukwila, Washington

Chemical Parameter	Number of samples with non-detects	Number of samples with detects	Total number of sample results	Detection frequency (%)	Maximum detected value	Number of results exceeding SQS [2]	Number of results exceeding CSL [2]
Ionizable Organic Compounds (ppb)							
Phenol	63	27	90	30%	3100	4	3
2-Methylphenol	59	0	59	0%	–	–	–
4-Methylphenol	77	13	90	14%	210	–	–
2,4-Dimethylphenol	59	0	59	0%	–	–	–
Pentachlorophenol	84	3	87	3%	930	2	2
Benzoic acid	66	21	87	24%	2000	16	16
Benzyl alcohol	50	6	56	11%	20	–	–
Miscellaneous (ppb)							
Hexachlorobutadiene	55	1	56	2%	0.99	–	–
N-Nitrosodiphenylamine	51	5	56	9%	2.6	–	–
Dibenzofuran	65	22	87	25%	680	1	–
PCBs (ppb)							
Aroclor 1016	50	0	50	0%	–	–	–
Aroclor 1221	80	0	80	0%	–	–	–
Aroclor 1232	80	0	80	0%	–	–	–
Aroclor 1242	44	36	80	45%	820	–	–
Aroclor 1248	73	7	80	9%	100	–	–
Aroclor 1254	11	46	57	81%	300	–	–
Aroclor 1254/1260	1	23	24	96%	1,700	–	–
Aroclor 1260	14	43	57	75%	130	–	–
Aroclor 1262	35	0	35	0%	–	–	–
Aroclor 1268	35	0	35	0%	–	–	–
Total PCBs	8	84	92	91%	2,500	6	2
Pesticides (ppb)							
2,4'-DDD	8	0	8	0%	–	na	na
2,4'-DDE	8	0	8	0%	–	na	na
2,4'-DDT	7	1	8	13%	1.4	na	na
4,4'-DDD	10	7	17	41%	6.4	na	na
4,4'-DDE	10	7	17	41%	6.4	na	na
4,4'-DDT	8	9	17	53%	37	na	na
Total DDTs	8	9	17	53%	47	na	na
Aldrin	16	1	17	6%	0.014	na	na
alpha-BHC	10	1	11	9%	0.14	na	na
alpha-Chlordane	10	7	17	41%	1.5	na	na
alpha-Endosulfan	9	0	9	0%	–	na	na
beta-BHC	10	1	11	9%	0.087	na	na
beta-Chlordane	10	1	11	9%	2.4	na	na
beta-Endosulfan	10	1	11	9%	0.47	na	na
cis-Nonachlor	7	0	7	0%	–	na	na
delta-BHC	9	1	10	10%	0.081	na	na
Dieldrin	13	4	17	24%	2.3	na	na
Endosulfan	1	1	2	50%	0.11	na	na
Endosulfan sulfate	10	1	11	9%	0.84	na	na
Endrin	10	1	11	9%	9.1	na	na

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TABLE 1

SUMMARY OF HISTORIC SEDIMENT ANALYTICAL DATA [1]

Former Rhone-Poulenc Site
Tukwila, Washington

Chemical Parameter	Number of samples with non-detects	Number of samples with detects	Total number of sample results	Detection frequency (%)	Maximum detected value	Number of results exceeding SQS [2]	Number of results exceeding CSL [2]
Pesticides (ppb) (Continued)							
Endrin aldehyde	9	2	11	18%	14	na	na
Endrin ketone	10	1	11	9%	3.7	na	na
gamma-BHC	11	6	17	35%	0.22	na	na
Heptachlor	16	1	17	6%	0.12	na	na
Heptachlor epoxide	10	1	11	9%	1	na	na
Methoxychlor	9	2	11	18%	10	na	na
Mirex	8	0	8	0%	--	na	na
Oxychlordane	7	0	7	0%	--	na	na
Total aldrin/dieldrin	13	4	17	24%	2.3	na	na
Total chlordane	10	7	17	41%	2.6	na	na
Toxaphene	9	0	9	0%	--	na	na
trans-Nonachlor	7	0	7	0%	--	na	na
Additional Metals (ppm)							
Aluminum	0	56	56	100%	29,000	na	na
Antimony	21	8	29	28%	5	na	na
Barium	0	56	56	100%	101	na	na
Beryllium	0	55	55	100%	0.57	na	na
Calcium	0	56	56	100%	7350	na	na
Cobalt	0	65	65	100%	12	na	na
Iron	0	56	56	100%	41600	na	na
Magnesium	0	56	56	100%	9640	na	na
Manganese	0	56	56	100%	886	na	na
Molybdenum	2	8	10	80%	2	na	na
Potassium	0	56	56	100%	3600	na	na
Selenium	11	18	29	62%	26	na	na
Sodium	0	56	56	100%	17800	na	na
Thallium	10	13	23	57%	0.13	na	na
Tin	11	3	14	21%	6	na	na
Vanadium	0	71	71	100%	89.6	na	na

Notes
1. Historic sediment analytical results from the Lower Duwamish Waterway Group Feasibility Dataset (AECOM, 2010)
2. "--" = no sample exceeded the indicated criterion for that analyte.
   na = No SQS or CSL criterion is established for the analyte.

Abbreviations
CSL = Cleanup Screening Level cited in the SMS.
HPAHs = high-moleclar weight polycyclic aromatic hydrocarbons
LPAHs = low-molecular-weight polycyclic aromatic hydrocarbons
PCBs = polychlorinated biphenyls
ppb = parts-per-billion
ppm = parts-per-million
SMS = Sediment Management Standards
SQS = Sediment Quality Standard
SVOCs = Semivolatile organic compounds

TABLE 2

SHORELINE INVESTIGATION SCREENING LEVELS AND LABORATORY REPORTING LIMITS
Former Rhone-Poulenc Site
Tukwila, Washington

soil concentrations in milligrams per kilogram (mg/kg)

Analyte[1]	Soil							
	Detection Limit [2,3]	Reporting Limit [2,3]	MTCA Method A, Unrestricted Land Use[4]	MTCA Method B, Carcinogen[4]	MTCA Method B, Noncarcinogen[4]	Puget Sound Soil Natural Background [5]	EPA RSL Industrial Soil Level[6]	EPA Soil Screening Level for Risk-Based Protection of Groundwater[6]
Metals for Soil								
Aluminum	3.55	5.0	--[9]	--	80,000	32,600	990,000	55,000
Arsenic	0.068	0.5	20	0.67	24	7.0	1.6	1.3E-03
Cadmium	0.11	0.2	2	--	80	1.0	800	1.4
Chromium	0.27	0.5	2,000	--	120,000	48	--	180,000
Copper	0.05	0.2	--	--	3,200	36	41,000	46
Lead	0.13	2.0	250	--	--	24	800	14
Mercury	0.0013	0.025	2	--	--	0.07	310	3.0E-02
Nickel	0.3	1.0	--	--	1,600	48	--	48
Selenium	0.65	5.0	--	--	400	--	--	9.5E-01
Thallium	0.53	5.0	--	--	--	--	--	--
Vanadium	0.06	0.3	--	--	5.6	--	72	2.6
Zinc	0.12	1.0	--	--	24,000	85	310,000	680
TPH for Soil								
TPH - Diesel range	0.742	5	2,000	--	--	--		
TPH - Heavy oil range	1.31	10	2,000	--	--	--		
TPH - Gasoline range	2.39	5	30[7]	--	--	--		
SVOCs for Soil								
1-Methylnaphthalene	0.00267	0.02	--	35	--	--	99	1.2E-02
1,2-Dichlorobenzene	0.00296	0.02	--	--	--	--	9,800	3.6E-01
1,3-Dichlorobenzene	0.00266	0.02	--	--	--	--	--	--
1,4-Dichlorobenzene	0.00273	0.02	--	--	--	--	12	4.1E-04
2,2-Oxybis(1-chloropropane)	0.00295	0.02	--	--	--	--	--	--
2,4,5-Trichlorophenol	0.0211	0.1	--	--	8,000	--	62,000	14
2,4,6-Trichlorophenol	0.0114	0.1	--	91	80	--	160	2.3E-02
2,4-Dichlorophenol	0.0183	0.1	--	--	240	--	1,800	1.3E-01
2,4-Dimethylphenol	0.00798	0.02	--	--	1,600	--	12,000	8.6E-01
2,4-Dinitrophenol	0.0499	0.2	--	--	160	--	1,200	8.2E-02
2,4-Dinitrotoluene	0.0194	0.1	--	--	160	--	5.5	2.9E-04
2,6-Dinitrotoluene	0.0151	0.1	--	--	80	--	620	5.0E-02
2-Chloronaphthalene	0.00292	0.02	--	--	6,400	--	82,000	15
2-Chlorophenol	0.00469	0.02	--	--	400	--	5,100	1.5E-01
2-Methylnaphthalene	0.00299	0.02	--	--	320	--	4,100	7.5E-01
2-Methylphenol	0.00534	0.02	--	--	4,000	--	--	--
2-Nitroaniline	0.0187	0.1	--	--	800	--	6,000	1.5E-01
2-Nitrophenol	0.00949	0.02	--	--	--	--	--	--
3-Nitroaniline	0.0252	0.1	--	--	--	--	--	--
3,3'-Dichlorobenzidine	0.0543	0.1	--	2.2	--	--	3.80	9.8E-04
4,6-Dinitro-2-methylphenol	0.0412	0.2	--	--	--	--	--	--
4-Bromophenyl phenyl ether	0.00379	0.02	--	--	--	--	--	--
4-Chloro-3-methylphenol	0.0152	0.1	--	--	--	--	--	--
4-Chloroaniline	0.0242	0.02	--	5.0	320	--	8.6	1.4E-04

TABLE 2

SHORELINE INVESTIGATION SCREENING LEVELS AND LABORATORY REPORTING LIMITS
Former Rhone-Poulenc Site
Tukwila, Washington

soil concentrations in milligrams per kilogram (mg/kg)

Analyte[1]	Soil							
	Detection Limit [2,3]	Reporting Limit [2,3]	MTCA Method A, Unrestricted Land Use[4]	MTCA Method B, Carcinogen[4]	MTCA Method B, Noncarcinogen[4]	Puget Sound Soil Natural Background [5]	EPA RSL Industrial Soil Level[6]	EPA Soil Screening Level for Risk-Based Protection of Groundwater[6]
SVOCs for Soil (Continued)								
4-Chlorophenyl phenyl ether	0.00296	0.02	--	--	--	--	--	--
4-Methylphenol (p-cresol)	0.00482	0.02	--	--	400	--	3,100	1.5
4-Nitroaniline	0.0229	0.1	--	--	--	--	86	1.4E-03
4-Nitrophenol	0.0281	0.1	--	--	--	--	--	--
Acenaphthene	0.0033	0.02	--	--	4,800	--	33,000	22
Acenaphthylene	0.003	0.02	--	--	--	--	--	--
Aniline	0.00181	0.02	--	180	560	--	300	4.0E-03
Anthracene	0.00437	0.02		--	24,000	--	170,000	360
Benzidine	NA	0.02	--	0.0043	240	--	7.5E-03	--
Benzo(a)anthracene	0.00462	0.02	*(8)*	1.4	--	--	2.1	1.0E-02
Benzo(a)pyrene	0.00513	0.02	0.10 [8]	0.14 [8]	--	--	0.21	3.5E-03
Benzo(ghi)perylene	0.00476	0.02	--	--	--	--	--	--
Total Benzofluoranthenes	0.0057	0.02	*(8)*	14 [8]	--	--	21	--
Benzoic acid	0.0426	0.2	--	--	320,000	--	2,500,000	34
Benzyl alcohol	0.0461	0.1	--	--	8,000	--	62,000	8.9E-01
Bis(2-chloroethoxy)methane	NA	0.02	--	--	--	--	1,800	2.5E-02
Bis-(2-chloroethyl) ether	0.00527	0.02	--	0.91	--	--	1.0	3.1E-06
Bis(2-ethylhexyl) phthalate	0.00873	0.02	--	71	1,600	--	120	1.10
Butyl benzyl phthalate	0.00411	0.02	--	530.0	16,000	--	910	5.1E-01
Carbazole	0.00238	0.02	--	--	--	--	--	--
Chrysene	0.00582	0.02	*(8)*	140 [8]	--	--	210	1.1
Dibenzo(a,h)anthracene	0.00454	0.02	*(8)*	0.14 [8]	--	--	0.0062	1.1E-02
Dibenzofuran	0.00315	0.02	--	--	80	--	--	--
Diethyl phthalate	0.00375	0.02	--	--	64,000	--	490,000	12
Dimethyl phthalate	0.00372	0.02	--	--	--	--	--	--
Di-n-butyl phthalate	0.00468	0.02	--	--	8,000	--	--	--
Di-n-octyl phthalate	0.00522	0.02	--	--	--	--	--	--
Fluoranthene	0.00438	0.02	--	--	3,200	--	22,000	160
Fluorene	0.00357	0.02	--	--	3,200	--	22,000	27
Hexachlorobenzene	0.00338	0.02	--	0.63	64	--	1.1	1.3E-02
Hexachlorobutadiene	0.0029	0.02	--	13	80	--	22	1.7E-03
Hexachlorocyclopentadiene	0.0124	0.1	--	--	480	--	3,700	6.8E-01
Hexachloroethane	0.00487	0.02	--	71	80	--	120	2.9E-03
Indeno(1,2,3-cd)pyrene	0.00505	0.02	*(8)*	14 [8]	--	--	2.1	1.2E-01
Isophorone	0.00269	0.02	--	1,100	16,000	--	1,800	2.3E-02
Naphthalene	0.00271	0.02	5.00	--	1,600	--	18	4.7E-04
Nitrobenzene	0.00376	0.02	--	--	160	--	24	7.9E-05
N-Nitrosodimethylamine	0.0144	0.02	--	0.02	0.64	--	3.4E-02	1.0E-07
N-Nitrosodi-n-propylamine	0.00283	0.02	--	0.14	--	--	2.5E-01	7.2E-06
N-Nitrosodiphenylamine	0.0128	0.02	--	200	--	--	350	7.5E-02
Pentachlorophenol	0.0274	0.1	--	2.5	400	--	9.0	5.7E-03

TABLE 2

SHORELINE INVESTIGATION SCREENING LEVELS AND LABORATORY REPORTING LIMITS
Former Rhone-Poulenc Site
Tukwila, Washington

soil concentrations in milligrams per kilogram (mg/kg)

Analyte[1]	Soil							
	Detection Limit [2,3]	Reporting Limit [2,3]	MTCA Method A, Unrestricted Land Use[4]	MTCA Method B, Carcinogen[4]	MTCA Method B, Noncarcinogen[4]	Puget Sound Soil Natural Background [5]	EPA RSL Industrial Soil Level[6]	EPA Soil Screening Level for Risk-Based Protection of Groundwater[6]
SVOCs for Soil (Continued)								
Phenanthrene	0.00361	0.02	--	--	--	--	--	--
Phenol	0.0038	0.02	--	--	24,000	--	180,000	6.3
Pyrene	0.00478	0.02	--	--	2,400	--	17,000	120
Pyridine	0.017	0.1	--	--	80	--	1,000	1.3E-02
VOCs for Soil								
1,1,1,2-Tetrachloroethane	0.000365	0.0005	--	38	2,400	--	9	2.0E-04
1,1,1-Trichloroethane	0.000151	0.0005	2.0	--	160,000	--	38,000	7.0E-02
1,1,2,2-Tetrachloroethane	0.00027	0.0005	--	5.0	1,600	--	--	--
trifluoroethane	0.000244	0.001	--	--	2,400,000	--	180,000	150.00
1,1,2-Trichloroethane	0.00023	0.0005	--	18	320	--	5	7.8E-05
1,1-Dichloroethane	0.000228	0.0005	--	--	16,000	--	2	6.9E-04
1,1-Dichloroethene	0.000258	0.0005	--	--	4,000	--	1,100	1.2E-01
1,1-Dichloropropene	0.000346	0.0005	--	--	--	--	--	--
1,2,3-Trichlorobenzene	0.000613	0.0025	--	--	--	--	490	8.7E-02
1,2,3-Trichloropropane	0.002666	0.001	--	0.03	320	--	9.5E-02	3.1E-07
1,2,4-Trichlorobenzene	0.000688	0.0025	--	--	400	--	99	6.8E-03
1,2,4-Trimethylbenzene	0.000301	0.0005	--	--	--	--	260	2.1E-02
1,2-Dibromo-3-chloropropane	0.000806	0.0025	--	1.30	16	--	6.9E-02	8.6E-05
1,2-Dibromoethane	0.000285	0.0005	0.01	--	--	--	1.7E-01	1.4E-05
1,2-Dichlorobenzene	0.00027	0.0005	--	--	7,200	--	9,800	5.8E-01
1,2-Dichloroethane	0.000194	0.0005	--	11.0	1,600	--	2	1.4E-03
1,2-Dichloropropane	0.000257	0.0005	--	--	--	--	5	1.7E-03
1,3,5-Trimethylbenzene	0.000364	0.0005	--	--	800	--	10,000	5.2E-01
1,3-Dichlorobenzene	0.000332	0.0005	--	--	--	--	--	--
1,3-Dichloropropane	0.000325	0.0005	--	--	--	--	20,000	2.5E-01
1,4-Dichlorobenzene	0.000365	0.0005	--	--	--	--	12	7.2E-02
2-Butanone	0.001071	0.0025	--	--	48,000	--	200,000	1.50
2-Chloroethyl vinyl ether	0.000835	0.0025	--	--	--	--	--	--
2-Chlorotoluene	0.000349	0.0005	--	--	--	--	20,000	7.1E-01
2,2-Dichloropropane	0.000386	0.0005	--	--	--	--	--	--
2-Hexanone	0.000265	0.0025	--	--	--	--	1,400	1.1E-02
4-Chlorotoluene	0.000403	0.0005	--	--	--	--	72,000	2.50
4-Methyl-2-pentanone	0.002144	0.0025	--	--	6,400	--	--	--
Acetone	0.00234	0.0025	--	--	72,000	--	630,000	4.50
Acrolein	0.001489	0.025	--	--	40	--	6.5E-01	8.4E-06
Acrylonitrile	0.000186	0.0025	--	1.90	--	--	1	9.9E-06
Benzene	0.000178	0.0005	0.03	18.0	320	--	5	2.6E-03
Bromobenzene	NA	0.0005	--	--	--	--	1,800	5.9E-02
Bromochloromethane	0.000241	0.0005	--	12.0	1,600	--	--	--
Bromodichloromethane	0.000244	0.0005	--	16.0	1,600	--	1	3.2E-05

TABLE 2

SHORELINE INVESTIGATION SCREENING LEVELS AND LABORATORY REPORTING LIMITS
Former Rhone-Poulenc Site
Tukwila, Washington

soil concentrations in milligrams per kilogram (mg/kg)

Analyte[1]	Soil							
	Detection Limit [2,3]	Reporting Limit [2,3]	MTCA Method A, Unrestricted Land Use[4]	MTCA Method B, Carcinogen[4]	MTCA Method B, Noncarcinogen[4]	Puget Sound Soil Natural Background [5]	EPA RSL Industrial Soil Level[6]	EPA Soil Screening Level for Risk-Based Protection of Groundwater[6]
VOCs for Soil (Continued)								
Bromoethane	0.00015	0.001	--	--	--	--	--	--
Bromoform	0.000274	0.0005	--	130	1,600	--	220	2.3E-03
Bromomethane	0.00051	0.001	--	--	110	--	32	2.2E-03
Carbon disulfide	0.000156	0.0005	--	--	8,000	--	3,700	3.1E-01
Carbon tetrachloride	NA	0.0005	--	14.3	320	--	3	1.7E-04
Chlorobenzene	0.000236	0.0005	--	--	1,600	--	1,400	6.8E-02
Chloroethane	0.000305	0.0005	--	--	--	--	--	--
Chloroform	0.000192	0.0005	--	--	800	--	2	2.2E-02
Chloromethane	0.000247	0.0005	--	--	--	--	500	4.9E-02
cis-1,2-Dichloroethene	0.000234	0.0005	--	--	160	--	10,000	2.1E-02
cis-1,3-Dichloropropene	0.000268	0.0005	--	--	--	--	10,000	2.1E-02
Dibromochloromethane	0.000254	0.0005	--	11.9	1,600	--	3	2.1E-02
Dibromomethane	0.000372	0.0005	--	--	--	--	110	2.0E-03
Dichlorodifluoromethane	0.000306	0.0005	--	--	16,000	--	780	6.1E-01
Ethylbenzene	0.000231	0.0005	6.0	--	8,000	--	27	7.8E-01
Hexachlorobutadiene	0.000579	0.0025	--	13.0	80	--	22	1.7E-03
Iodomethane	0.000294	0.0005	--	--	--	--	--	--
Isopropylbenzene (cumene)	0.0003	0.0005	--	--	8,000	--	11,000	1.10
m,p-Xylenes	0.000551	0.0005	9.0	--	16,000	--	2,700	9.80
Methyl tert-butyl ether	0.000309	0.0005	0.10	--	--	--	220	2.8E-03
Methylene chloride	0.000357	0.001	0.02	130	4,800	--	53	1.3E-03
n-Butylbenzene	0.000449	0.001	--	--	--	--	--	--
n-Propylbenzene	0.000321	0.0005	--	--	8,000	--	21,000	2.50
o-Xylene	0.000284	0.0005	--	--	16,000	--	19,000	1.20
p-Isopropyltoluene	0.000382	0.0005	--	--	--	--	--	--
sec-Butylbenzene	0.000377	0.0005	--	--	--	--	--	--
Styrene	0.00031	0.0005	--	--	16,000	--	36,000	1.80
tert-Butylbenzene	0.000345	0.0005	--	--	--	--	--	--
Tetrachloroethene	0.00023	0.0005	0.05	1.90	800	--	3	2.3E-03
Toluene	0.000457	0.0005	7.0	--	6,400	--	45,000	6.9E-01
trans-1,2-Dichloroethene	0.000239	0.0005	--	--	1,600	--	690	2.9E-02
trans-1,3-Dichloropropene	0.000284	0.0005	--	--	--	--	--	--
Trichloroethene	0.000168	0.0005	0.03	11.0	1,050	--	14	1.8E-03
Trichlorofluoromethane	0.000193	0.0005	--	--	24,000	--	3,400	8.3E-01
Vinyl chloride	0.000251	0.0005	--	--	240	--	2	6.9E-04
Vinyl acetate	0.000238	0.0025	--	--	--	--	4,100	8.8E-02

TABLE 2

SHORELINE INVESTIGATION SCREENING LEVELS AND LABORATORY REPORTING LIMITS

Former Rhone-Poulenc Site

Tukwila, Washington

soil concentrations in milligrams per kilogram (mg/kg)

Analyte[1]	Soil							
	Detection Limit [2,3]	Reporting Limit [2,3]	MTCA Method A, Unrestricted Land Use[4]	MTCA Method B, Carcinogen[4]	MTCA Method B, Noncarcinogen[4]	Puget Sound Soil Natural Background [5]	EPA RSL Industrial Soil Level[6]	EPA Soil Screening Level for Risk-Based Protection of Groundwater[5]
PCBs for Soil								
Aroclor 1016	0.577	0.004	--	14.0	5.60	--	21.00	9.2E-02
Aroclor 1221	--	0.004	--	--	--	--	5.4E-01	1.2E-04
Aroclor 1232	--	0.004	--	--	--	--	5.4E-01	1.2E-04
Aroclor 1242	--	0.004	--	--	--	--	7.4E-01	5.3E-03
Aroclor 1248	--	0.004	--	--	--	--	7.4E-01	5.2E-03
Aroclor 1254	--	0.004	--	0.50	1.60	--	7.4E-01	8.8E-03
Aroclor 1260	0.61	0.004	--	0.50	--	--	7.4E-01	2.4E-02

TABLE 2

SHORELINE INVESTIGATION SCREENING LEVELS AND LABORATORY REPORTING LIMITS

Former Rhone-Poulenc Site

Tukwila, Washington

groundwater concentrations in micrograms per liter (µg/L)

Analyte[1]	Groundwater														
	Detection Limit [2,3]	Reporting Limit [2,3]	Groundwater MTCA Method A[4] (µg/L)	Groundwater MTCA Method B, Carcinogen[4] (µg/L)	Groundwater MTCA Method B, Non-Carcinogen[4] (µg/L)	Surface Water - Aquatic Life - Fresh/Acute-Ch.173-201A WAC (µg/L)	Surface Water - Aquatic Life - Fresh/Acute-Clean Water Act 304 (µg/L)	Surface Water ARAR - Aquatic Life - Fresh/Acute - National Toxics Rule - 40 CFR 131 (µg/L)	Surface Water ARAR - Aquatic Life - Fresh/Chronic - Ch. 173-201A WAC (µg/L)	Surface Water ARAR - Aquatic Life - Fresh/Chronic - Clean Water Act §304 (µg/L)	Surface Water ARAR - Aquatic Life - Fresh/Chronic - National Toxics Rule, 40 CFR 131 (µg/L)	Surface Water ARAR - Human Health – Fresh Water – Clean Water Act §304 (µg/L)	Surface Water ARAR - Human Health – Fresh Water – National Toxics Rule, 40 CFR 131 (µg/L)	Surface Water MTCA Method B, Carcinogen[4] (µg/L)	Surface Water MTCA Method B, Non-Carcinogen[4] (µg/L)
Metals for Groundwater															
Aluminum	25.67	50.00	--	--	--	--	--	--	--	--	--	--	--	--	--
Arsenic	3.33	50.00	5	0.058333	4.8	360.00	340.00	360.00	190.00	150.00	190.00	0.02	0.02	0.10	17.68
Cadmium	0.18	2	5	--	16	0.82	2	3.9	0.37	0.25	1	--	--	--	40.50
Chromium	1.24	5	50	--	--	--	--	--	--	--	--	--	--	--	--
Copper	0.92	2	--	--	640	4.61	13	17	3.47	9	11	--	--	--	2,880.00
Lead	1.55	1.0	15.0	--	--	13.9	65.0	65.0	0.5	2.5	2.5	--	--	--	--
Mercury	0.0026	0.02	2	--	--	2.1	1.4	2.1	0.012	0.77	0.012	--	0.14	--	--
Nickel	3.86	10	--	--	--	--	--	--	--	--	--	--	--	--	--
Selenium	4.99	50	--	--	--	--	--	--	--	--	--	--	--	--	--
Thallium	1.41	50	--	--	--	--	--	--	--	--	--	--	--	--	--
Vanadium	0.27	3	--	--	1.12	--	--	--	--	--	--	--	--	--	--
Zinc	1.45	10.0	--	--	4800	35.4	120.0	110.0	32.3	120.0	100.0	7,400.00	--	--	16,548.46
TPH for Groundwater															
TPH - Diesel range	16	100	500	--	--	--	--	--	--	--	--	2,000.00	--	--	N/A
TPH - Heavy oil range	49	200	500	--	--	--	--	--	--	--	--	2,000.00	--	--	N/A
TPH - Gasoline range	60	250	800 [7]	--	--	--	--	--	--	--	--	30.00	--	--	N/A
SVOCs for Groundwater															
1-Methylnaphthalene	0.479	1	--	1.51	--	--	--	--	--	--	--	--	--	--	--
1,2-Dichlorobenzene	0.365	1	--	--	--	--	--	--	--	--	--	--	--	--	--
1,3-Dichlorobenzene	0.358	1	--	--	--	--	--	--	--	--	--	--	--	--	--
1,4-Dichlorobenzene	0.397	1	--	--	--	--	--	--	--	--	--	--	--	--	--
2,2-Oxybis(1-chloropropane)	0.623	1	--	--	--	--	--	--	--	--	--	1,400.00	1,400.00	--	--
2,4,5-Trichlorophenol	2.22	5	--	--	800	--	--	--	--	--	--	1,800.00	--	--	--
2,4,6-Trichlorophenol	2.408	5	--	3.977272727	8	--	--	--	--	--	--	1.40	2.10	3.93	17.30
2,4-Dichlorophenol	2.597	5	--	--	24	--	--	--	--	--	--	77.00	93.00	--	191.10
2,4-Dimethylphenol	0.359	1	--	--	160	--	--	--	--	--	--	380.00	--	--	552.79
2,4-Dinitrophenol	3.48	10	--	--	32	--	--	--	--	--	--	69.00	70.00	--	3,456.79
2,4-Dinitrotoluene	2.52	5	--	--	32	--	--	--	--	--	--	0.11	0.11	--	1,364.52
2,6-Dinitrotoluene	2.393	5	--	--	16	--	--	--	--	--	--	--	--	--	--
2-Chloronaphthalene	0.477	1	--	--	640	--	--	--	--	--	--	1,000.00	--	--	1,026.77
2-Chlorophenol	0.529	1	--	--	40	--	--	--	--	--	--	--	--	--	96.74
2-Methylnaphthalene	0.475	1	--	--	32	--	--	--	--	--	--	--	--	--	--
2-Methylphenol	0.531	1	--	--	400	--	--	--	--	--	--	--	--	--	--
2-Nitroaniline	2.627	5	--	--	160	--	--	--	--	--	--	--	--	--	--
2-Nitrophenol	1.968	5	--	--	--	--	--	--	--	--	--	--	--	--	--
3-Nitroaniline	2.314	5	--	--	--	--	--	--	--	--	--	--	--	--	--
3,3'-Dichlorobenzidine	1.51	5	--	0.194444444	--	--	--	--	--	--	--	0.02	0.04	0.05	--
4,6-Dinitro-2-methylphenol	3.087	10	--	--	--	--	--	--	--	--	--	--	--	--	--
4-Bromophenyl phenyl ether	0.423	1	--	--	--	--	--	--	--	--	--	--	--	--	--
4-Chloro-3-methylphenol	2.417	5	--	--	--	--	--	--	--	--	--	--	--	--	--
4-Chloroaniline	2.599	5	--	0.219	32	--	--	--	--	--	--	--	--	--	--

TABLE 2

SHORELINE INVESTIGATION SCREENING LEVELS AND LABORATORY REPORTING LIMITS

Former Rhone-Poulenc Site

Tukwila, Washington

groundwater concentrations in micrograms per liter (µg/L)

Analyte[1]	Groundwater														
	Detection Limit [2,3]	Reporting Limit [2,3]	Groundwater MTCA Method A[4] (µg/L)	Groundwater MTCA Method B, Carcinogen[4] (µg/L)	Groundwater MTCA Method B, Non-Carcinogen[4] (µg/L)	Surface Water - Aquatic Life - Fresh/Acute-Ch.173-201A WAC (µg/L)	Surface Water - Aquatic Life - Fresh/Acute-Clean Water Act 304 (µg/L)	Surface Water ARAR - Aquatic Life - Fresh/Acute - National Toxics Rule - 40 CFR 131 (µg/L)	Surface Water ARAR - Aquatic Life - Fresh/Chronic - Ch. 173-201A WAC (µg/L)	Surface Water ARAR - Aquatic Life - Fresh/Chronic - Clean Water Act §304 (µg/L)	Surface Water ARAR - Aquatic Life - Fresh/Chronic - National Toxics Rule, 40 CFR 131 (µg/L)	Surface Water ARAR - Human Health – Fresh Water – Clean Water Act §304 (µg/L)	Surface Water ARAR - Human Health – Fresh Water – National Toxics Rule, 40 CFR 131 (µg/L)	Surface Water MTCA Method B, Carcinogen[4] (µg/L)	Surface Water MTCA Method B, Non-Carcinogen[4] (µg/L)
SVOCs for Groundwater (Continued)															
4-Chlorophenyl phenyl ether	0.451	1	–	–	–	–	–	–	–	–	–	–	–	–	–
4-Methylphenol (p-cresol)	0.523	1	–	–	40	–	–	–	–	–	–	–	–	–	–
4-Nitroaniline	2.249	5	–	–	–	–	–	–	–	–	–	–	–	–	–
4-Nitrophenol	2.573	5	–	–	–	–	–	–	–	–	–	–	–	–	–
Acenaphthene	0.546	1	–	–	960	–	–	–	–	–	–	670.00	–	–	642.79
Acenaphthylene	0.48	1	–	–	–	–	–	–	–	–	–	–	–	–	–
Aniline	0.255	1	–	7.675438596	56	–	–	–	–	–	–	–	–	–	–
Anthracene	0.531	1	–	–	4800	–	–	–	–	–	–	8,300.00	9,600.00	–	25,925.93
Benzidine	–	10	–	0.000380435	48	–	–	–	–	–	–	0.00	0.0001	0.00	88.8888889
Benzo(a)anthracene	0.52	1	–	0.12	–	–	–	–	–	–	–	0.00	0.00	0.30	–
Benzo(a)pyrene	0.484	1	0.1	0.011986301	–	–	–	–	–	–	–	0.00	0.00	0.03	–
Benzo(ghi)perylene	0.546	1	–	–	–	–	–	–	–	–	–	–	–	–	–
Total Benzofluoranthenes	0.483	1	–	–	–	–	–	–	–	–	–	–	–	–	–
Benzoic acid	5.111	10	–	–	64000	–	–	–	–	–	–	–	–	–	–
Benzyl alcohol	2.008	5	–	–	800	–	–	–	–	–	–	–	–	–	–
Bis(2-chloroethoxy)methane	0.565	1	–	–	–	–	–	–	–	–	–	–	–	–	–
Bis-(2-chloroethyl) ether	0.583	1	–	0.039772727	–	–	–	–	–	–	–	0.03	0.03	0.85	–
Bis(2-ethylhexyl) phthalate	1.877	1	–	6.25	320	–	–	–	–	–	–	1.20	1.80	3.56	398.86
Butyl benzyl phthalate	0.557	1	–	46.1	3200	–	–	–	–	–	–	1,500.00	–	8.24	1,250.00
Carbazole	0.306	1	–	–	–	–	–	–	–	–	–	–	–	–	–
Chrysene	0.549	1	–	12	–	–	–	–	–	–	–	0.00	0.00	29.60	–
Dibenzo(a,h)anthracene	0.482	1	–	0.012	–	–	–	–	–	–	–	0.00	0.00	0.03	–
Dibenzofuran	0.479	1	–	–	16	–	–	–	–	–	–	–	–	–	–
Diethyl phthalate	0.582	1	–	–	240	–	–	–	–	–	–	17,000.00	23,000.00	–	–
Dimethyl phthalate	0.528	1	–	–	–	–	–	–	–	–	–	270,000.00	313,000.00	–	–
Di-n-butyl phthalate	0.537	1	–	–	1600	–	–	–	–	–	–	2,000.00	2,700.00	–	2,913.03
Di-n-octyl phthalate	0.508	1	–	–	–	–	–	–	–	–	–	–	–	–	–
Fluoranthene	0.515	1	–	–	640	–	–	–	–	–	–	130.00	300.00	–	90.18
Fluorene	0.558	1	–	–	640	–	–	–	–	–	–	1,100.00	1,300.00	–	3,456.79
Hexachlorobenzene	0.47	1	–	0.0546875	12.8	–	–	–	–	–	–	0.00	0.00	0.00	0.2386737
Hexachlorobutadiene	0.306	1	–	0.560897436	8	–	–	–	–	–	–	0.44	0.44	29.89	933.00
Hexachlorocyclopentadiene	1.181	5	–	–	48	–	–	–	–	–	–	40.00	240.00	–	3,584.23
Hexachloroethane	0.35	1	–	3.125	8	–	–	–	–	–	–	1.40	1.90	5.33	29.83
Indeno(1,2,3-cd)pyrene	0.485	1	–	0.12	–	–	–	–	–	–	–	0.00	0.00	0.30	–
Isophorone	0.481	1	–	46.05263158	1600	–	–	–	–	–	–	35.00	8.40	1,557.67	118,383.22
Naphthalene	0.522	1	160	–	160	–	–	–	–	–	–	–	–	–	4,938.27
Nitrobenzene	0.575	1	–	–	16	–	–	–	–	–	–	17.00	17.00	–	1,790.00
N-Nitrosodimethylamine	2.648	5	–	0.000857843	0.064	–	–	–	–	–	–	0.00	0.00	4.89	798.00
N-Nitrosodi-n-propylamine	0.56	1.000	–	–	–	–	–	–	–	–	–	0.01	–	0.82	–
N-Nitrosodiphenylamine	0.46	1	–	–	–	–	–	–	–	–	–	3.30	5.00	9.73	–
Pentachlorophenol	2.411	5	–	0.219	80	20.27	19	20	12.79	15	13	0.27	0.28	1.47	1,180.00

TABLE 2

SHORELINE INVESTIGATION SCREENING LEVELS AND LABORATORY REPORTING LIMITS

Former Rhone-Poulenc Site

Tukwila, Washington

groundwater concentrations in micrograms per liter (µg/L)

Analyte[1]	Groundwater														
	Detection Limit [2,3]	Reporting Limit [2,3]	Groundwater MTCA Method A[4] (µg/L)	Groundwater MTCA Method B, Carcinogen[4] (µg/L)	Groundwater MTCA Method B, Non-Carcinogen[4] (µg/L)	Surface Water - Aquatic Life - Fresh/Acute-Ch.173-201A WAC (µg/L)	Surface Water - Aquatic Life - Fresh/Acute-Clean Water Act 304 (µg/L)	Surface Water ARAR - Aquatic Life - Fresh/Acute - National Toxics Rule - 40 CFR 131 (µg/L)	Surface Water ARAR - Aquatic Life - Fresh/Chronic - Ch. 173-201A WAC (µg/L)	Surface Water ARAR - Aquatic Life - Fresh/Chronic - Clean Water Act §304 (µg/L)	Surface Water ARAR - Aquatic Life - Fresh/Chronic - National Toxics Rule, 40 CFR 131 (µg/L)	Surface Water ARAR - Human Health – Fresh Water – Clean Water Act §304 (µg/L)	Surface Water ARAR - Human Health – Fresh Water – National Toxics Rule, 40 CFR 131 (µg/L)	Surface Water MTCA Method B, Carcinogen[4] (µg/L)	Surface Water MTCA Method B, Non-Carcinogen[4] (µg/L)
SVOCs for Groundwater (Continued)															
Phenanthrene	0.557	1	--	--	--	--	--	--	--	--	--	--	--	--	--
Phenol	0.519	1	--	--	2400	--	--	--	--	--	--	21,000.00	21,000.00	--	556,000.00
Pyrene	0.547	1	--	--	480	--	--	--	--	--	--	830.00	960.00	--	2,592.59
Pyridine	--	5	--	--	8	--	--	--	--	--	--	--	--	--	--
VOCs for Groundwater															
1,1,1,2-Tetrachloroethane	0.068	0.2	--	1.682692308	240	--	--	--	--	--	--	--	--	--	--
1,1,1-Trichloroethane	0.089	0.2	200	--	16000	--	--	--	--	--	--	--	--	--	926,000.00
1,1,2,2-Tetrachloroethane	0.067	0.2	--	0.21875	160	--	--	--	--	--	--	0.17	0.17	6.48	10,400.00
trifluoroethane	0.107	0.2	--	--	240,000.00	--	--	--	--	--	--	--	--	--	--
1,1,2-Trichloroethane	0.035	0.2	--	0.76754386	32	--	--	--	--	--	--	0.59	0.60	25.27	2,304.53
1,1-Dichloroethane	0.053	0.2	5.00	0.48	160	--	--	--	--	--	--	--	--	59.35	43,200.00
1,1-Dichloroethene	0.091	0.2	--	--	1,600.00	--	--	--	--	--	--	330.00	0.06	--	--
1,1-Dichloropropene	0.092	0.2	--	--	--	--	--	--	--	--	--	--	--	--	--
1,2,3-Trichlorobenzene	0.087	0.5	--	--	--	--	--	--	--	--	--	--	--	--	--
1,2,3-Trichloropropane	0.226	0.5	--	0.00146	32.00	--	--	--	--	--	--	--	--	--	--
1,2,4-Trichlorobenzene	0.1	0.5	--	1.51	80	--	--	--	--	--	--	35.00	--	1.96	227.42
1,2,4-Trimethylbenzene	0.058	0.2	--	--	--	--	--	--	--	--	--	--	--	--	--
1,2-Dibromo-3-chloropropane	0.212	0.5	--	0.031	--	--	--	--	--	--	--	--	--	--	--
1,2-Dibromoethane	0.075	0.2	--	0.05	1.6	--	--	--	--	--	--	--	--	--	--
1,2-Dichlorobenzene	0.055	0.2	--	--	720	--	--	--	--	--	--	420.00	2,700.00	--	4,196.64
1,2-Dichloroethane	0.075	0.2	5.00	0.480769231	160	--	--	--	--	--	--	0.38	0.38	59.35	43,200.00
1,2-Dichloropropane	0.093	0.2	--	--	--	--	--	--	--	--	--	0.50	--	--	--
1,3,5-Trimethylbenzene	0.063	0.2	--	--	80	--	--	--	--	--	--	--	--	--	--
1,3-Dichlorobenzene	0.04	0.2	--	--	--	--	--	--	--	--	--	320.00	400.00	--	--
1,3-Dichloropropane	0.02	0.2	--	--	--	--	--	--	--	--	--	--	--	--	--
1,4-Dichlorobenzene	0.057	0.2	--	--	--	--	--	--	--	--	--	63.00	400.00	--	--
2-Butanone	0.808	5	--	--	4800	--	--	--	--	--	--	--	--	--	--
2-Chloroethyl vinyl ether	0.086	1	--	--	--	--	--	--	--	--	--	--	--	--	--
2-Chlorotoluene	0.042	0.2	--	--	160	--	--	--	--	--	--	--	--	--	--
2,2-Dichloropropane	0.083	0.2	--	--	--	--	--	--	--	--	--	--	--	--	--
2-Hexanone	0.31	5	--	--	--	--	--	--	--	--	--	--	--	--	--
4-Chlorotoluene	0.073	0.2	--	--	--	--	--	--	--	--	--	--	--	--	--
4-Methyl-2-pentanone	0.384	5	--	--	640	--	--	--	--	--	--	--	--	--	--
Acetone	0.72	5	--	--	7200	--	--	--	--	--	--	--	--	--	--
Acrolein	0.292	5	--	--	4.00	--	--	--	--	--	--	190.00	320.00	--	--
Acrylonitrile	0.185	1	--	0.081018519	--	--	--	--	--	--	--	0.05	0.06	0.40	--
Benzene	0.056	0.2	5	0.795454545	32	--	--	--	--	--	--	2.20	1.20	22.66	1,990.00
Bromobenzene	0.051	0.2	--	--	--	--	--	--	--	--	--	--	--	--	--
Bromochloromethane	0.067	0.2	--	0.52	160.00	--	--	--	--	--	--	0.40	0.41	21.00	14,000.00
Bromodichloromethane	0.053	0.2	--	0.705645161	160	--	--	--	--	--	--	0.55	0.27	27.88	13,827.16

TABLE 2

SHORELINE INVESTIGATION SCREENING LEVELS AND LABORATORY REPORTING LIMITS
Former Rhone-Poulenc Site
Tukwila, Washington

groundwater concentrations in micrograms per liter (µg/L)

Analyte[1]	Groundwater														
	Detection Limit [2,3]	Reporting Limit [2,3]	Groundwater MTCA Method A[4] (µg/L)	Groundwater MTCA Method B, Carcinogen[4] (µg/L)	Groundwater MTCA Method B, Non-Carcinogen[4] (µg/L)	Surface Water - Aquatic Life - Fresh/Acute-Ch.173-201A WAC (µg/L)	Surface Water - Aquatic Life - Fresh/Acute-Clean Water Act 304 (µg/L)	Surface Water ARAR - Aquatic Life - Fresh/Acute - National Toxics Rule - 40 CFR 131 (µg/L)	Surface Water ARAR - Aquatic Life - Fresh/Chronic - Ch. 173-201A WAC (µg/L)	Surface Water ARAR - Aquatic Life - Fresh/Chronic - Clean Water Act §304 (µg/L)	Surface Water ARAR - Aquatic Life - Fresh/Chronic - National Toxics Rule, 40 CFR 131 (µg/L)	Surface Water ARAR - Human Health – Fresh Water – Clean Water Act §304 (µg/L)	Surface Water ARAR - Human Health – Fresh Water – National Toxics Rule, 40 CFR 131 (µg/L)	Surface Water MTCA Method B, Carcinogen[4] (µg/L)	Surface Water MTCA Method B, Non-Carcinogen[4] (µg/L)
VOCs for Groundwater (Continued)															
Bromoethane	0.09	1	--	--	--	--	--	--	--	--	--	--	--	--	--
Bromoform	0.07	0.2	--	5.537974684	160	--	--	--	--	--	--	4.30	4.30	218.78	13,827.16
Bromomethane	0.043	0.2	--	--	11.2	--	--	--	--	--	--	47.00	48.00	--	967.90
Carbon disulfide	0.087	0.2	--	--	800	--	--	--	--	--	--	--	--	--	--
Carbon tetrachloride	0.075	0.2	--	0.625	32	--	--	--	--	--	--	0.23	0.25	4.94	553.00
Chlorobenzene	0.042	0.2	--	--	160	--	--	--	--	--	--	130.00	680.00	--	5,034.16
Chloroethane	0.152	0.2	--	--	--	--	--	--	--	--	--	--	--	--	--
Chloroform	0.081	0.2	--	--	80	--	--	--	--	--	--	5.70	5.70	--	6,913.58
Chloromethane	0.098	0.2	--	--	--	--	--	--	--	--	--	--	--	--	--
cis-1,2-Dichloroethene	0.1	0.2	--	--	16.00	--	--	--	--	--	--	--	--	--	--
cis-1,3-Dichloropropene	0.058	0.2	--	--	--	--	--	--	--	--	--	--	--	--	--
Dibromochloromethane	0.09	0.2	--	0.52	160.00	--	--	--	--	--	--	0.40	0.41	20.58	13,827.16
Dibromomethane	0.081	0.2	--	--	--	--	--	--	--	--	--	--	--	--	--
Dichlorodifluoromethane	0.084	0.2	--	--	1600	--	--	--	--	--	--	--	--	--	--
Ethylbenzene	0.094	0.2	700	--	800	--	--	--	--	--	--	530.00	3,100.00	--	6,913.58
Hexachlorobutadiene	0.112	0.5	--	0.56	8	--	--	--	--	--	--	0.44	0.44	29.89	933.00
Iodomethane	0.04	0.2	--	--	--	--	--	--	--	--	--	--	--	--	--
Isopropylbenzene (cumene)	0.062	0.2	--	--	800	--	--	--	--	--	--	--	--	--	--
m,p-Xylenes	0.144	0.4	1000	--	1600	--	--	--	--	--	--	--	--	--	--
Methyl tert-butyl ether	0.046	0.5	20.00	--	--	--	--	--	--	--	--	--	--	--	--
Methylene chloride	0.391	0.2	5.00	5.833333333	480	--	--	--	--	--	--	4.60	4.70	960.22	172,839.51
n-Butylbenzene	0.108	0.2	--	--	--	--	--	--	--	--	--	--	--	--	--
n-Propylbenzene	0.081	0.2	--	--	--	--	--	--	--	--	--	--	--	--	--
o-Xylene	0.057	0.2	--	--	1600	--	--	--	--	--	--	--	--	--	--
p-Isopropyltoluene	0.075	0.2	--	--	--	--	--	--	--	--	--	--	--	--	--
sec-Butylbenzene	0.077	0.2	--	--	--	--	--	--	--	--	--	--	--	--	--
Styrene	0.066	0.2	--	--	1600	--	--	--	--	--	--	--	--	--	--
tert-Butylbenzene	0.061	0.2	--	--	--	--	--	--	--	--	--	--	--	--	--
Tetrachloroethene	0.088	0.2	5	see additional information	80	--	--	--	--	--	--	0.69	0.80	--	836
Toluene	0.056	0.2	1000	--	640	--	--	--	--	--	--	1,300	6,800	--	19,400
trans-1,2-Dichloroethene	0.085	0.2	--	--	160	--	--	--	--	--	--	140,000	--	--	32,817.63
trans-1,3-Dichloropropene	0.059	0.2	--	--	--	--	--	--	--	--	--	--	--	--	--
Trichloroethene	0.076	0.2	5	see additional information	see additional information	--	--	--	--	--	--	2.50	2.70	--	--
Trichlorofluoromethane	0.092	0.2	--	--	2,400	--	--	--	--	--	--	--	--	--	--
Vinyl chloride	0.075	0.2	0.2	see additional information	24	--	--	--	--	--	--	0.03	2.00	see additional information	6,647.67
Vinyl acetate	0.068	1	--	--	--	--	--	--	--	--	--	--	--	--	--

TABLE 2

SHORELINE INVESTIGATION SCREENING LEVELS AND LABORATORY REPORTING LIMITS

Former Rhone-Poulenc Site

Tukwila, Washington

groundwater concentrations in micrograms per liter (µg/L)

Analyte[1]	Groundwater														
	Detection Limit [2,3]	Reporting Limit [2,3]	Groundwater MTCA Method A[4] (µg/L)	Groundwater MTCA Method B, Carcinogen[4] (µg/L)	Groundwater MTCA Method B, Non-Carcinogen[4] (µg/L)	Surface Water - Aquatic Life - Fresh/Acute-Ch.173-201A WAC (µg/L)	Surface Water - Aquatic Life - Fresh/Acute-Clean Water Act 304 (µg/L)	Surface Water ARAR - Aquatic Life - Fresh/Acute - National Toxics Rule - 40 CFR 131 (µg/L)	Surface Water ARAR - Aquatic Life - Fresh/Chronic - Ch. 173-201A WAC (µg/L)	Surface Water ARAR - Aquatic Life - Fresh/Chronic - Clean Water Act §304 (µg/L)	Surface Water ARAR - Aquatic Life - Fresh/Chronic - National Toxics Rule, 40 CFR 131 (µg/L)	Surface Water ARAR - Human Health – Fresh Water – Clean Water Act §304 (µg/L)	Surface Water ARAR - Human Health – Fresh Water – National Toxics Rule, 40 CFR 131 (µg/L)	Surface Water MTCA Method B, Carcinogen[4] (µg/L)	Surface Water MTCA Method B, Non-Carcinogen[4] (µg/L)
PCBs for Groundwater															
Aroclor 1016	0.00248	0.01	--	1.25	1.12	--	--	--	--	--	0.014	--	--	0.00	0.01
Aroclor 1221	--	0.01	--	--	--	--	--	--	--	--	--	--	--	--	--
Aroclor 1232	--	0.01	--	--	--	--	--	--	--	--	--	--	--	--	--
Aroclor 1242	--	0.01	--	--	--	--	--	--	--	--	--	--	--	--	--
Aroclor 1248	--	0.01	--	--	--	--	--	--	--	--	--	--	--	--	--
Aroclor 1254	--	0.01	--	0.0437	0.32	--	--	--	--	--	0.014	--	--	0.00	0.0016619
Aroclor 1260	0.00276	0.01	--	0.0437	--	--	--	--	--	--	0.014	--	--	--	--

Notes

1. Samples will be analyzed using the methods listed in Tables 4 and/or 5.
2. In order to achieve reporting limits below the screening levels, the laboratory will report detections between the detection limit and the reporting limit. In the event that the detection limit for non-detected results is greater than the screening level, the laboratory will evaluate options for reporting lower detection limits, such as increasing volume during extraction or analyzing using select ion monitoring. Usability of non-detected results that are greater than the screening levels will be evaluated on a case-by-case basis.
3. Reporting and detection limits established by ARI and obtained from www.arilabs.com. Reporting limits based on wet weight and will be slightly higher on a dry weight basis, including matrix interference.
4. MTCA Method A and Method B cleanup levels based on Ecology's Model Toxics Control Act (MTCA) regulations and obtained from Ecology's CLARC database.
5. Puget Sound background concentrations as listed in Washington State Department of Ecology Publication (Ecology, 1994).
6. Values obtained from EPA Regional Screening Levels (EPA, 2011).
7. Screening level for gasoline if benzene is also present in the sample.
8. A total value for all carcinogenic polycyclic aromatic hydrocarbon compounds adjusted for toxicity equivalents [WAC-173-340-708(8)] is calculated and compared to the screening level for benzo(a)pyrene.
9. -- = No value available.

Abbreviations

ARAR = Applicable or Relevant and Appropriate Requirements
CFR = Code of Federal Regulations
CLARC = Cleanup Levels and Risk Calculation
MTCA = Model Toxics Control Act
PCBs = polychlorinated biphenyls
RSL = Regional Screening Level
SVOCs = semivolatile organic compounds
TPH = total petroleum hydrocarbons
VOCs = volatile organic compounds
WAC = Washington Administrative Code

amec

TABLE 3

SHORELINE INVESTIGATION SAMPLING MATRIX

Former Rhone-Poulenc Site
Tukwila, Washington

Investigation Focus	Number of Sample Locations	Matrix Types	Sample Depth(s) [1] (feet)	Analytes
Southwest Corner and Slip No. 6	6 locations	Soil	0.5 to 2.0	Metals[2], VOCs[3], and pH[4]
			5.0 to 7.0	
			10.0 to 12.0	
			15.0 to 17.0	
			20.0 to 22.0	
			25.0 to 27.0	
			30.0 to 32.0	
			35.0 to 37.0	
	3 locations		40.0 to 42.0	
			45.0 to 47.0	
			50.0 to 52.0	
			55.0 to 57.0	
	6 locations	Groundwater	15	Metals[2], VOCs[3], and pH[4]
			20	
			25	
			30	
			34	
	3 locations (additional depths)		40	
			45	
			50	
			55	
	4 to 6 locations[5]		varies	Additional analyses will include cations[6], anions[7], sulfide[8], ammonia[9], alkalinity[10], and specific gravity[11]
	2 locations	Pore Water	3	Metals[2], VOCs[3], and pH[4]
Duwamish Riverbank	6 locations	Soil	0.5 to 2.0	Metals[2], TPH-G[12], TPH-Dx[13], VOCs[3], SVOCs[14] and PCBs[15]. Up to three samples will be selected in the field and analyzed for VPH and EPH[16].
			5.0 to 7.0	
			10.0 to 12.0	
			15.0 to 17.0	
Geotechnical Analyses	3 Southwest Corner and 3 Duwamish Riverbank Locations	Soil	Various depths	Selected soil samples will be analyzed for sieve grain size and/or hydrometer analyses[17], moisture[18], Atterberg limits[19], and density[20].

Notes

1. Sample depths listed are approximate; actual sample depths will depend on sample recovery and the specific tooling available on the driect-push drill rig.
2. Metals include Al, As, Cd, Cr, Cu, Pb, Hg, Ni, Se, Th, V, and Zn. Soil and water samples will be analyzed using EPA Method 6010B except for mercury, which will be analyzed using EPA Method 7470A for water and 7471A for soil.
3. VOCs will be analyzed using EPA 8260C.
4. pH in water will be measured using EPA 150.1 and pH in soil will be measured using EPA 9045D.
5. The exact sample locations and depths will be determined in the field based on pH readings and spatial variability.
6. Cations include Ca, Mg, Na, K, and Si and will be analyzed using EPA Method 200.8.
7. Anions include Cl^-, NO_3^-, SO_4^{-2}, and PO_4^{-3} and will be analyzed using EPA Method 300.0.

TABLE 3

SHORELINE INVESTIGATION SAMPLING MATRIX
Former Rhone-Poulenc Site
Tukwila, Washington

Notes (Continued)
8. Sulfide will be analyzed using Standard Method (SM) 4500.
9. Ammonia will be analyzed using EPA Method 350.1.
10. Alkalinity will be analyzed using SM 2320.
11. Specific gravity of liquids will be analyzed using ASTM D1298.
12. TPH-G will be analyzed using NWTPH method.
13. TPH-Dx will be analyzed using NWTPH method.
14. SVOCs will include pentachlorophenol and will be analyzed using EPA Method 8270D.
15. PCBs will be analyzed using EPA Method 8082.
16. VPH will be analyzed using Ecology WADOE-VPH/EPA 5035; EPH using Ecology WADOE-EPH.
17. Sieve grain size analyses will be performed using ASTM D421; hydrometer analyses will be performed using ASTM D422.
18. Moisture will be determined using ASTM D2216.
19. Atterberg limits will be determined using ASTM D4318.
20. Density will be determined using ASTM D2937/API RP40/EPA 9100.

Abbreviations
EPA = U.S. Environmental Protection Agency
EPH = Extractable Petroleum Hydrocarbons
PCBs = Polychlorinated biphenyls
SVOCs = Semivolatile organic compounds
TDS = Total Dissolved Solids
TPH-Dx = Total Petroleum Hydrocarbons - Diesel Range Extended
TPH-G = Total Petroleum Hydrocarbons - Gasoline Range
VOCs = Volatile Organic Compounds
VPH = Volatile Petroleum Hydrocarbons

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TABLE 4

SOIL SAMPLE CONTAINERS, PRESERVATION, AND HOLDING TIMES

Former Rhone-Poulenc Site
Tukwila, Washington

Analyte		Analytical Method[1]	Sample Container	Preservation Temperature	Holding Time[2]
Conventionals	pH	EPA 9045D	4 oz. wide-mouth glass jar	NA	14 days
Metals	Mercury	EPA 7471A	4 oz. wide-mouth glass jar	≤ 6°C	28 days
	Arsenic	EPA 200.8			6 months
	All other compounds	EPA 6010B			
TPH	Total petroleum hydrocarbons (TPH) - diesel and heavy oil range	Ecology NWTPH-Dx	4 oz. wide-mouth glass jar	≤ 6°C	14 days[4]
	TPH - Gasoline	Ecology NWTPH-Gx/EPA 5035	2 x 40 mL vial	≤ 6°C / MeOH	14 days if preserved w/ MeOH, 2 days if unpreserved
	EPH	Ecology WADOE-EPH	8 oz. wide-mouth glass jar	≤ 6°C	14 days [4]
	VPH	Ecology WADOE-VPH/EPA 5035 [5]	2 x 40 mL vial (no headspace)	≤ 6°C / MeOH	14 days if preserved w/ MeOH, 2 days if unpreserved
Semivolatile organic compounds (SVOCs)		EPA 8270D	8 oz. wide-mouth glass jar	≤ 6°C	14 days[4]
Volatile organic compounds (VOCs)		EPA 8260C/ EPA 5035	4 x 40 mL vial (no headspace)	≤ 6°C	14 days
Polychlorinated biphenyls (PCBs)		EPA 8082 low level	8 oz. wide-mouth glass jar	≤ 6°C	14 days[4]
Geotechnical Analyses	Grain size (sieve and/or hydrometer)	ASTM D421/422	4 oz. plastic bag	NA	NA
	Moisture content	ASTM D2216	2 oz. plastic bag	NA	NA
	Atterberg limits	ASTM D4318	4 oz. plastic bag (less than 425 µm)	NA	NA
	Bulk Density	ASTM D2937/API RP40/EPA 9100	4 oz. core or bag	NA	NA

Notes
1. Method numbers refer to EPA SW-846 Analytical Methods, Washington State Department of Ecology (WADOE) analytical methods, or ASTM International (ASTM) standards.
2. Holding times are based on elapsed time from date of collection.
3. Metals include Al, Cd, Cr, Cu, Pb, Hg, Ni, Se, Th, V, and Zn.
4. Holding time is 14 days to extraction and 40 days from extraction to analysis.
5. Samples collected using method 5035 should also include a 4 oz. jar of soil for total solids determination.

Abbreviations
°C = degrees Celcius
µm = micrometer
MeOH = methanol
mL = milliliter
NA = not applicable
oz = ounce

TABLE 5

GROUNDWATER SAMPLE CONTAINERS, PRESERVATION, AND HOLDING TIMES

Former Rhone-Poulenc Site
Tukwila, Washington

Analyte		Analytical Method[1]	Sample Container	Preservation Temperature	Holding Time[2]
Conventionals	pH	EPA 150.1	500 mL HDPE	NA	Analyze in the field or immediately upon receipt
	Alkalinity	SM 2320	500 mL HDPE (no headspace)	≤ 6°C	14 days
	Anions [3]	EPA 300.0	500 mL HDPE	≤ 6°C	48 hours
	Ammonia	EPA 350.1	500 mL HDPE	pH < 2 with 9N H_2SO_4; cool to ≤ 6°C	28 days
	Sulfide	SM 4500	500 mL HDPE (no headspace)	pH > 9 with 1N zinc acetate + 1 mL 10N NaOH; cool to ≤ 6°C	7 days
	specific gravity of liquids	ASTM D1298	500 mL HDPE	≤ 6°C	7 days
Total Metals[4]	Mercury	EPA 7470A	500 mL HDPE	5 mL 1:1 HNO_3	28 days
	Arsenic	EPA 200.8	500 mL HDPE	2.5 mL 1:1 HNO_3	6 months
	Cations [5]	EPA 6010B			
	All other constituents	EPA 6010B			
Volatile organic compounds (VOCs)		EPA 8260C	3 x 40 mL vial (no headspace)	≤ 6°C	7 days [6]

Notes

1. Method numbers refer to EPA SW-846 Analytical Methods or Standard Methods for the Examination of Water and Wastewater (SM) or Methods for the Chemical Analysis of Water and Wastes (MCAWW) (EPA/500/4-79/020)
2. Holding times are based on elapsed time from date of collection.
3. Anions include: chloride, sulfate, nitrate, and phosphorus. Phosphorus requires field filtration.
4. Metals include Al, Cd, Cr, Cu, Pb, Hg, Ni, Se, Th, V, and Zn.
5. Cations include Ca, Mg, Na, K, and Si.
6. Due to the possibility of sample effervescence upon preservation, VOC samples will not be preserved. Note the holding time of 7 days.

Abbreviations

°C = degrees Celcius
HDPE = high density polyethylene
L = liter
mL = milliliter
NA = not applicable
TDS = total dissolved solids

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TABLE 6

SHORELINE INVESTIGATION SUPPLEMENTAL QAPP REVISION TABLE

Former Rhone-Poulenc Site
Tukwila, Washington

Matrix	Analysis	Existing QAPP	Shoreline Investigation Method	Resolution
Soil	Metals	EPA 6010B with lead by EPA 7421.	EPA 6020/EPA 200.8	Samples will be analyzed using EPA Method 6020/200.8. SOP for method is attached in Appendix A.
		Analytes listed are Sb, As, Cd, Cr, Cu, Zn, Pb, Hg	Al, Ni, Se, Th, and V are included in addition to existing analytes	No changes required since all metals are covered by EPA Method 6020. SOP attached in Appendix A.
	VOCs	Method is EPA 8260B	EPA 8260C with EPA 5035	Samples will be collected following EPA Method 5035 procedures. The SOP is attached in Appendix A.
	Geotechnical Analyses	Geotechnical Analyses are not covered in the Soil QAPP	Grain size, moisture, Atterberg Limits, and density may be requested	SOPs for the new methods will be included in Appendix A.
Water	Metals	EPA 6000 series	EPA 200.8	Samples will be analyzed using EPA Method 200.8. SOP for method is attached in Appendix A.
		EPA 7000 series	EPA 200.8	All metals will be analyzed using EPA 200.8. SOP is attached in Appendix A.
		Analytes listed are As, Cd, Ca, Cr, Cu, Fe, Mg, Pb, Mn, Ni, K, Se, Th, Na, V, Zn	Al is included in addition to existing analytes	No changes required since all metals are covered by EPA Method 200.8.
	pH	Not listed in Groundwater QAPP	EPA 150.1	No changes required since SOP in Appendix of Soil QAPP includes the water method.
	VOCs	EPA 8260B and EPA 8021	EPA 8060C	The SOP for EPA Method 8260C is attached in Appendix A.

Abbreviations

Al = aluminum
As = arsenic
Ca = calcium
Cd = cadmium
Cr = chromium
Cu = copper
EPA = U.S. Environmental Protection Agency
Fe = iron
Hg = mercury
K = potassium
Mg = magnesium
Mn = manganese
Na = sodium
Ni = nickel
Pb = lead
QAPP = quality assurance project plan
Sb = antimony
Se = selenium
SOP = standard operating procedure
Th = thorium
V = vanadium
VOCs = volatile organic compounds
Zn = zinc

TABLE 7

SEDIMENT SAMPLE LOCATIONS

Former Rhone-Poulenc Site
Tukwila, Washington

Proposed Sample Location	State Plane Coordinates (WA SPC North NAD 83; Survey Feet)		Estimated Mudline Elevation (feet MLLW) [1]
	Easting	Northing	
RP-01	1276587	193530	5.3
RP-02	1276518	193463	2.7
RP-03	1276611	193436	4.2
RP-04 [2]	1276541	193369	2.7
RP-21	1276541	193369	2.7
RP-05	1276634	193342	4.3
RP-06	1276565	193275	2.8
RP-07	1276657	193249	4.7
RP-08	1276588	193182	2.7
RP-09	1276681	193155	5.6
RP-10 [3]	1276611	193088	2.5
RP-22	1276611	193088	2.5
RP-11	1276704	193061	5.8
RP-12	1276635	192994	2.2
RP-13	1276728	192968	5.6
RP-14	1276658	192900	2.4
RP-15	1276751	192874	3.4
RP-16	1276681	192807	0.4
RP-17	1276770	192785	0.5
RP-18	1276750	192821	3
RP-19	1277205	192955	3
RP-20	1276907	192761	-3.5

Notes
1. Estimated from bathymetric survey.
2. Duplicate core collected at this location (sample designated RP-21).
3. Duplicate core collected at this location (sample designated RP-22).

Abbreviations
MLLW = mean lower low water
NAD = North American Datum
SPC = State Plane Coordinates

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TABLE 8

SEDIMENT ACTION LEVELS AND REPORTING LIMITS

Former Rhone-Poulenc Site
Tukwila, Washington

Analyte	Sediment Management Standards [1]		LAET [4]	Sediment DQOs [5]	Sample Preparation/ Extraction	Analytical Method	Method Detection Limit	Reporting Limit [6]
	SQS [2]	CSL [3]						
Metals (mg/kg)	mg/kg dry wt	mg/kg dry wt	mg/kg dry wt	mg/kg dry wt				
Arsenic	57	93	57	—	EPA 3050	EPA 200.8 (ICPMS)	0.068	0.5
Cadmium	5.1	6.7	5.1	—	EPA 3050	EPA 6010 (ICP-OES)	0.11	0.2
Chromium	260	270	260	—	EPA 3050	EPA 6010 (ICP-OES)	0.27	0.5
Copper	390	390	390	—	EPA 3050	EPA 6010 (ICP-OES)	0.05	0.2
Lead	450	530	450	—	EPA 3050	EPA 6010 (ICP-OES)	0.13	2.0
Mercury	0.41	0.59	0.41	—	EPA 7471A	EPA 7471A (CVAA)	0.0013	0.05
Silver	6.1	6.1	6.1	—	EPA 3050	EPA 6010 (ICP-OES)	0.03	1.0
Vanadium	—	—	—	0.3	EPA 3050	EPA 6010 (ICP-OES)	0.06	0.3
Zinc	410	960	410	—	EPA 3050	EPA 6010 (ICP-OES)	0.12	1.0
Nonionizable Organic Compounds	mg/kg carbon	mg/kg carbon	µg/kg dry wt	µg/kg dry wt				
Aromatic Hydrocarbons (µg/kg)								
Total LPAH	370	780	5,200	—	—	—	—	—
Naphthalene	99	170	2,100	—	EPA 3550B	EPA 8270D - PSEP	2.71	20
Acenaphthylene	66	66	1,300	—	EPA 3550B	EPA 8270D - PSEP	3.00	20
Acenaphthene	16	57	500	—	EPA 3550B	EPA 8270D - PSEP	3.30	20
Fluorene	23	79	540	—	EPA 3550B	EPA 8270D - PSEP	3.57	20
Phenanthrene	100	480	1,500	—	EPA 3550B	EPA 8270D - PSEP	3.61	20
Anthracene	220	1,200	960	—	EPA 3550B	EPA 8270D - PSEP	4.37	20
2-Methylnaphthalene	38	780	670	—	EPA 3550B	EPA 8270D - PSEP	2.99	20
Total HPAH	960	5,300	12,000	—	—	—	—	—
Fluoranthene	160	1,200	1,700	—	EPA 3550B	EPA 8270D - PSEP	4.38	20
Pyrene	1,000	1,400	2,600	—	EPA 3550B	EPA 8270D - PSEP	4.78	20
Benz[a]anthracene	110	270	1,300	—	EPA 3550B	EPA 8270D - PSEP	4.62	20

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TABLE 8

SEDIMENT ACTION LEVELS AND REPORTING LIMITS

Former Rhone-Poulenc Site
Tukwila, Washington

Analyte	Sediment Management Standards [1]		LAET [4]	Sediment DQOs [5]	Sample Preparation/ Extraction	Analytical Method	Method Detection Limit	Reporting Limit [6]
	SQS [2]	CSL [3]						
Chrysene	110	460	1,400	—	EPA 3550B	EPA 8270D - PSEP	5.82	20
Total benzofluoranthenes	230	450	3,200	—	EPA 3550B	EPA 8270D - PSEP	5.70	40
Benzo[a]pyrene	99	210	1,600	—	EPA 3550B	EPA 8270D - PSEP	5.13	20
Indeno[1,2,3-c,d]pyrene	34	88	600	—	EPA 3550B	EPA 8270D - PSEP	5.05	20
Dibenzo[a,h]anthracene	12	33	230	—	EPA 3550B	EPA 8270D - PSEP	4.54	20
Benzo[g,h,i]perylene	31	78	670	—	EPA 3550B	EPA 8270D - PSEP	4.76	20
Chlorinated Benzenes (µg/kg)								
1,2-Dichlorobenzene	2.3	2.3	35	—	EPA 3550B	EPA 8270D - PSEP	2.96	20
1,4-Dichlorobenzene	3.1	9	110	—	EPA 3550B	EPA 8270D - PSEP	2.73	20
1,2,4-Trichlorobenzene	0.81	1.8	31	—	EPA 3550B	EPA 8260C - PSEP	3.79	5
Hexachlorobenzene	0.38	2.3	22	—	EPA 3550B	EPA 8081A - PSEP	3.38	1
Phthalate Esters (µg/kg)								
Dimethyl phthalate	53	53	71	—	EPA 3550B	EPA 8270D - PSEP	3.72	20
Diethyl phthalate	61	110	200	—	EPA 3550B	EPA 8270D - PSEP	3.75	20
Di-n-butyl phthalate	220	1,700	1,400	—	EPA 3550B	EPA 8270D - PSEP	4.68	20
Butyl benzyl phthalate	4.9	64	63	—	EPA 3550B	EPA 8270D - PSEP	4.11	20
Bis[2-ethylhexyl] phthalate	47	78	1,300	—	EPA 3550B	EPA 8270D - PSEP	8.73	20
Di-n-octyl phthalate	58	4,500	6,200	—	EPA 3550B	EPA 8270D - PSEP	5.22	20
Miscellaneous (µg/kg)								
Dibenzofuran	15	58	540	—	EPA 3550B	EPA 8270D - PSEP	3.15	20
Hexachlorobutadiene	3.9	6.2	11	—	EPA 3550B	EPA 8081A - PSEP	0.138	0.5
N-Nitrosodiphenylamine	11	11	28	—	EPA 3550B	EPA 8270D - PSEP	12.8	20
Total PCBs	12	65	130	—	PSDDA Sonication [7] (low levels)	EPA 8082	9.33 to 10.82	20 µg/kg per Aroclor

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TABLE 8

SEDIMENT ACTION LEVELS AND REPORTING LIMITS

Former Rhone-Poulenc Site
Tukwila, Washington

Analyte	Sediment Management Standards [1]		LAET [4]	Sediment DQOs [5]	Sample Preparation/ Extraction	Analytical Method	Method Detection Limit	Reporting Limit [6]
	SQS [2]	CSL [3]						
Ionizable Organic Compounds (µg/kg)	**µg/kg dry wt**	**µg/kg dry wt**	**µg/kg dry wt**	**µg/kg dry wt**				
Phenol	420	1,200	420	—	EPA 3550B	EPA 8270D - PSEP	3.80	20
2-Methylphenol	63	63	63	—	EPA 3550B	EPA 8270D - PSEP	5.34	20
4-Methylphenol	670	670	670	—	EPA 3550B	EPA 8270D - PSEP	4.82	20
2,4-Dimethylphenol	29	29	29	—	EPA 3550B	EPA 8270D - PSEP	7.98	20
Pentachlorophenol	360	690	360	—	EPA 3550B	EPA 8270D - PSEP	27.4	100
Benzyl alcohol	57	73	57	—	EPA 3550B	EPA 8270D - PSEP	46.1	100
Benzoic acid	650	650	650	—	EPA 3550B	EPA 8270D - PSEP	42.6	200
Pesticides (µg/kg)	**µg/kg dry wt**	**µg/kg dry wt**	**µg/kg dry wt**	**µg/kg dry wt**				
Dieldrin	—	—	—	2	EPA 3550B	EPA 8081A - PSEP	0.100	1.0

Notes

1. SMS criteria for nonionizable organic compounds are expressed in mg/kg carbon, since SMS for most nonionizable organic compounds are generally expressed as a carbon-normalized value. However, values for nonionizable organic compounds are usually not carbon-normalized in sediments with TOC values above 4% or below 0.5%, and the so dry-weight equivalent (LAET) values for these constituents are also provided. In these cases, the dry weight equivalent values are generally used instead.
2. SQS = Sediment Quality Standards (WAC 173-204-320).
3. CSL = Cleanup Screening Levels (WAC 173-204-520).
4. LAET = Lowest Apparent Effects Threshold. Dry weight equivalent of the SMS "SQS."
5. Analytical laboratory reporting limits.
6. Reporting limits obtained from Analytical Resources, Inc., laboratory.
7. Puget Sound Dredged Disposal Analysis protocol for low detection limits.

TABLE 8

SEDIMENT ACTION LEVELS AND REPORTING LIMITS
Former Rhone-Poulenc Site
Tukwila, Washington

Analyte	Sediment Management Standards [1]		LAET [4]	Sediment DQOs [5]	Sample Preparation/ Extraction	Analytical Method	Method Detection Limit	Reporting Limit [6]
	SQS [2]	CSL [3]						

Abbreviations
µg/kg = micrograms per kilogram
ARI = Analytical Resources, Inc.
CSL = cleanup screening level
CVAA = cold-vapor atomic absorption
DQOs = data quality objectives
dry wt = dry weight
EPA = U.S. Environmental Protection Agency
HPAH = high-molecular-weight polycyclic aromatic hydrocarbons
ICP-MS = inductively coupled plasma/mass spectrometer
ICP-OES = inductively coupled plasma/optical emission spectrophotometer
LPAH = low-molecular-weight polycyclic aromatic hydrocarbons
mg/kg = milligrams per kilogram
mg/kg = milligrams per kilogram
PAH = polycyclic aromatic hydrocarbons
PCBs = polychlorinated biphenyls
PCBs = polychlorinated biphenyls
PSDDA = Puget Sound Dredged Disposal Analysis
PSEP = Puget Sound Estuary Program
SQS = Sediment Quality Standards
TOC = total organic carbon
WAC = Washington Administrative Code
wt = weight

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TABLE 9

SEDIMENT SAMPLING DEPTHS AND PROPOSED INITIAL ANALYTES

Former Rhone-Poulenc Site
Tukwila, Washington

Proposed Sample Location	Sample Type	Samples Collected [1]	Preliminary List of Initial Samples Analyzed [2]	Analyses
RP-01	Grab	top 10 cm	top 10 cm	SMS SQS, vanadium, dieldrin, TS, TOC, grain size
	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			4- to 5-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			8- to 9-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			12- to 13-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
RP-02	Grab	top 10 cm	top 10 cm	SMS SQS, vanadium, dieldrin, TS, TOC, grain size
	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			4- to 5-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			8- to 9-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			12- to 13-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
RP-03	Grab	top 10 cm	top 10 cm	SMS SQS, vanadium, dieldrin, TS, TOC, grain size
	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			4- to 5-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			8- to 9-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			12- to 13-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
RP-04 [3]	Grab	top 10 cm	top 10 cm	SMS SQS, vanadium, dieldrin, TS, TOC, grain size
	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			4- to 5-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			8- to 9-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			12- to 13-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
RP-21	Grab	top 10 cm	top 10 cm	SMS SQS, vanadium, dieldrin, TS, TOC, grain size
	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			4- to 5-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			8- to 9-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			12- to 13-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
RP-05	Grab	top 10 cm	top 10 cm	SMS SQS, vanadium, dieldrin, TS, TOC, grain size
	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			4- to 5-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			8- to 9-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			12- to 13-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
RP-06	Grab	top 10 cm	top 10 cm	SMS SQS, vanadium, dieldrin, TS, TOC, grain size
	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			4- to 5-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			8- to 9-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			12- to 13-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
RP-07	Grab	top 10 cm	top 10 cm	SMS SQS, vanadium, dieldrin, TS, TOC, grain size
	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			4- to 5-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			8- to 9-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			12- to 13-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
RP-08	Grab	top 10 cm	top 10 cm	SMS SQS, vanadium, dieldrin, TS, TOC, grain size
	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			4- to 5-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			8- to 9-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			12- to 13-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
RP-09	Grab	top 10 cm	top 10 cm	SMS SQS, vanadium, dieldrin, TS, TOC, grain size
	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			4- to 5-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			8- to 9-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			12- to 13-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC

TABLE 9

SEDIMENT SAMPLING DEPTHS AND PROPOSED INITIAL ANALYTES

Former Rhone-Poulenc Site
Tukwila, Washington

Proposed Sample Location	Sample Type	Samples Collected [1]	Preliminary List of Initial Samples Analyzed [2]	Analyses
RP-10 [4]	Grab	top 10 cm	top 10 cm	SMS SQS, vanadium, dieldrin, TS, TOC, grain size
	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			4- to 5-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			8- to 9-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			12- to 13-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
RP-22	Grab	top 10 cm	top 10 cm	SMS SQS, vanadium, dieldrin, TS, TOC, grain size
	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			4- to 5-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			8- to 9-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			(12- to 13-foot sample duplicate not required)	
RP-11	Grab	top 10 cm	top 10 cm	SMS SQS, vanadium, dieldrin, TS, TOC, grain size
	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			4- to 5-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			8- to 9-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			12- to 13-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
RP-12	Grab	top 10 cm	top 10 cm	SMS SQS, vanadium, dieldrin, TS, TOC, grain size
	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			4- to 5-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			8- to 9-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			12- to 13-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
RP-13	Grab	top 10 cm	top 10 cm	SMS SQS, vanadium, dieldrin, TS, TOC, grain size
	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			4- to 5-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			8- to 9-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			12- to 13-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
RP-14	Grab	top 10 cm	top 10 cm	SMS SQS, vanadium, dieldrin, TS, TOC, grain size
	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			4- to 5-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			8- to 9-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			12- to 13-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
RP-15	Grab	top 10 cm	top 10 cm	SMS SQS, vanadium, dieldrin, TS, TOC, grain size
	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			4- to 5-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			8- to 9-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			12- to 13-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
RP-16	Grab	top 10 cm	top 10 cm	SMS SQS, vanadium, dieldrin, TS, TOC, grain size
	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			4- to 5-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			8- to 9-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			12- to 13-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
RP-17	Grab	top 10 cm	top 10 cm	SMS SQS, vanadium, dieldrin, TS, TOC, grain size
	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			4- to 5-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			8- to 9-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			12- to 13-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
RP-18	Grab	top 10 cm	top 10 cm	SMS SQS, vanadium, dieldrin, TS, TOC, grain size
	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			4- to 5-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			8- to 9-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			12- to 13-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC

TABLE 9

SEDIMENT SAMPLING DEPTHS AND PROPOSED INITIAL ANALYTES

Former Rhone-Poulenc Site
Tukwila, Washington

Proposed Sample Location	Sample Type	Samples Collected [1]	Preliminary List of Initial Samples Analyzed [2]	Analyses
RP-19	Grab	top 10 cm	top 10 cm	SMS SQS, vanadium, dieldrin, TS, TOC, grain size
	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			4- to 5-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			8- to 9-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			12- to 13-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
RP-20	Grab	top 10 cm	top 10 cm	SMS SQS, vanadium, dieldrin, TS, TOC, grain size
	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			4- to 5-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			8- to 9-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC
			12- to 13-foot interval	SMS SQS, vanadium, dieldrin, TS, TOC

Notes

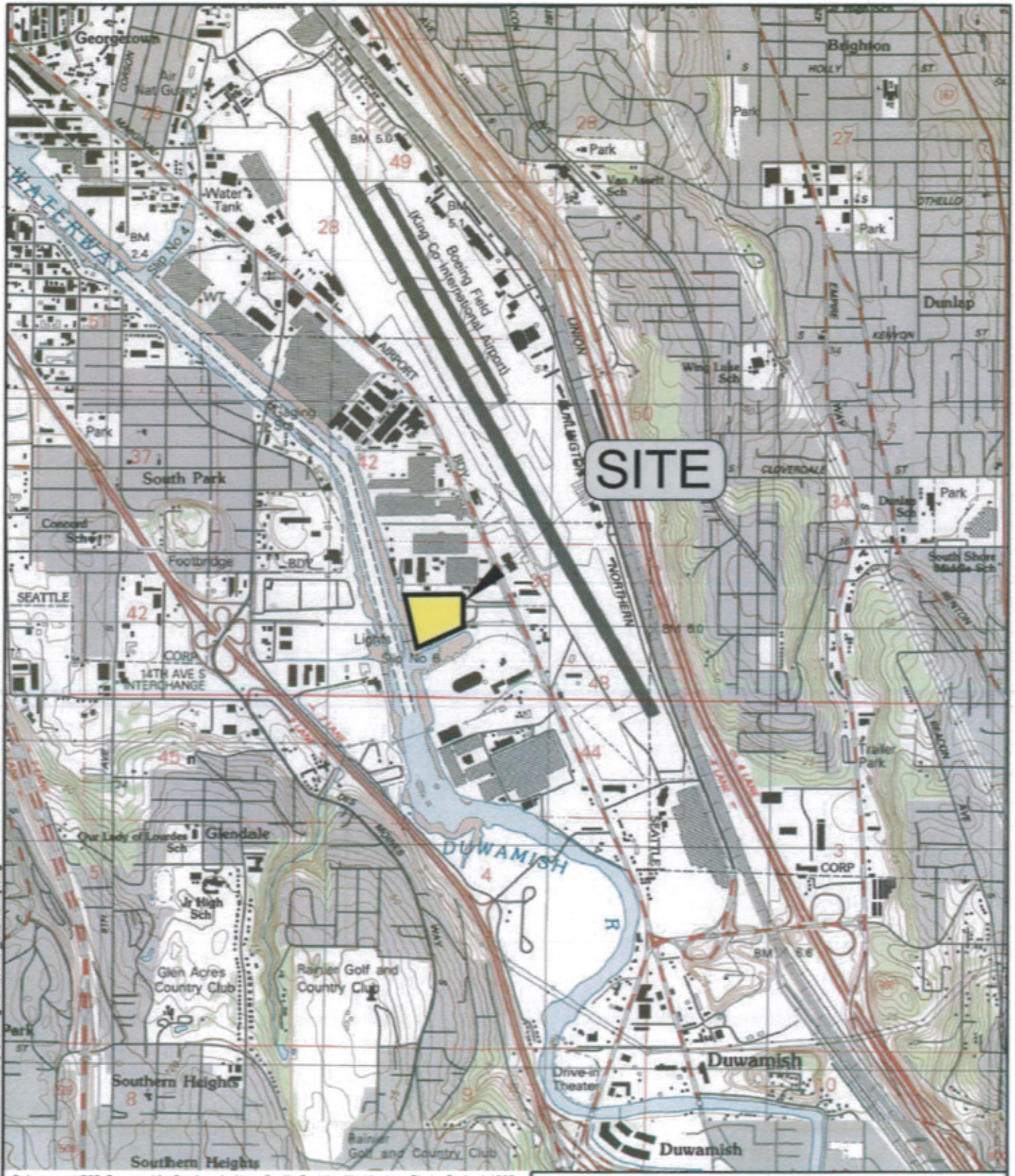
1. See Section 3.2.1 for discussion of the target sample depth.
2. Intervals to be analyzed may be changed based on sediment characteristics observed by the field geologist during core processing and actual recovery depth.
3. Duplicate core collected at this location (sample designated RP-21).
4. Duplicate core collected at this location (sample designated RP-22).

Abbreviations

SMS = Sediment Management Standards
SQS = Sediment Quality Standards
TOC = Total Organic Carbon
TS = Total Solids

FIGURES

FIGURES



Reference: USGS Topographic Quadrangle Map, South Seattle, Washington, Photo Revised 1968

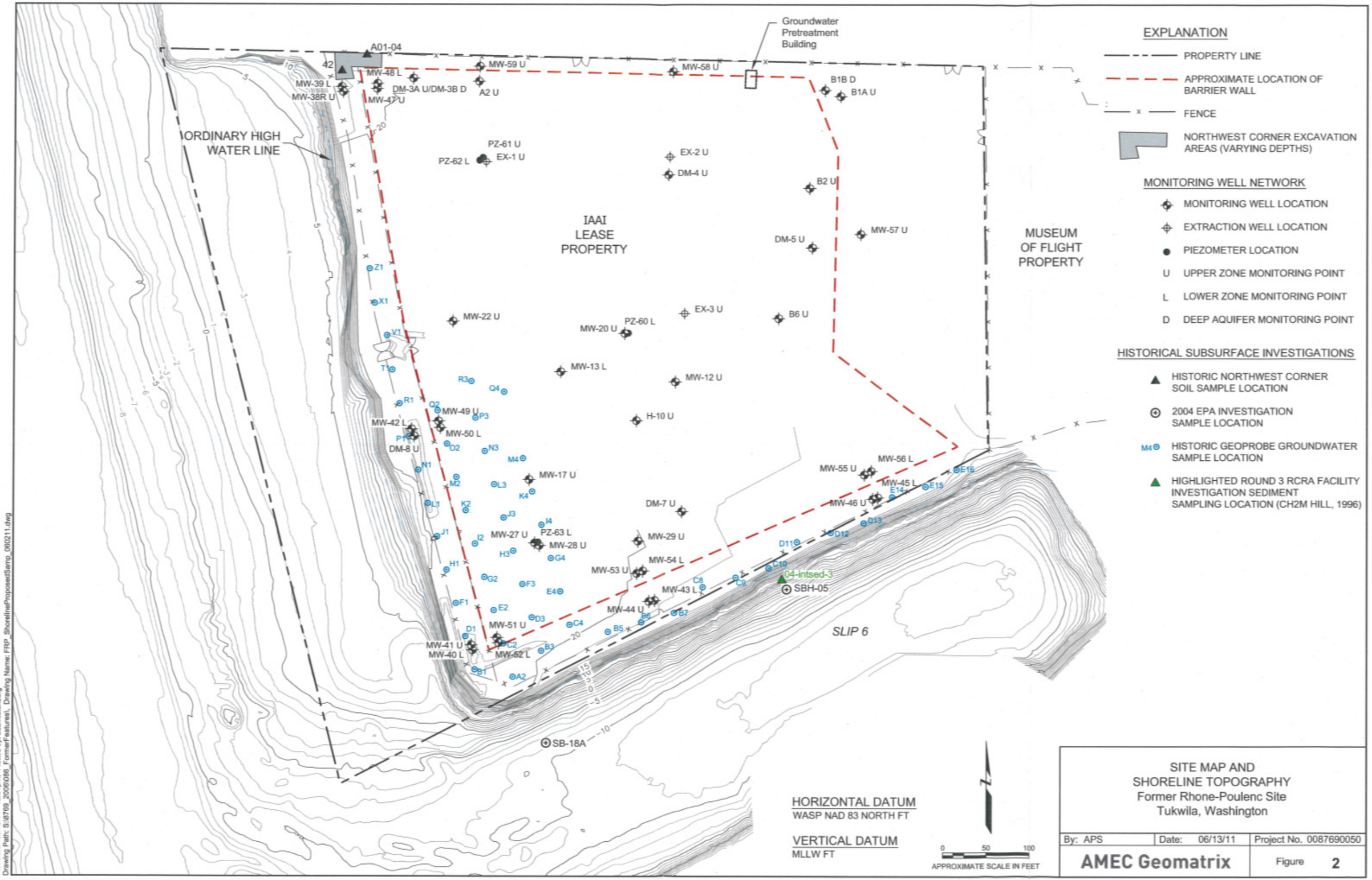
0 1000 2000

APPROXIMATE SCALE IN FEET

SITE VICINITY MAP
Former Rhone-Poulenc Site
Tukwila, Washington

By: APS	Date: 11/08/10	Project No. 0087690050
AMEC Geomatrix		Figure 1

Plot Date: 11/08/10 - 12:52pm, Plotted by: adam.stenberg
Drawing Path: S:\87690_2006\079_ShorelineSediment\PICAD\, Drawing Name: FRP_SiteVicinity.dwg



SITE MAP AND
SHORELINE TOPOGRAPHY
Former Rhone-Poulenc Site
Tukwila, Washington

By: APS	Date: 06/13/11	Project No. 0087690050
AMEC Geomatrix		Figure 2

Plot Date: 06/13/11 - 12:04pm, Plotted by: adam.sternberg
Drawing Path: S:\8769_2006\086_FormerFeatures\, Drawing Name: FRP_ShorelineProposedSamp_060211.dwg

HAZARDOUS WASTE STORAGE AREA (HWSA)
CATCH BASIN LOCATION
MW-59 U
MW-48 L
MW-39 L
MW-38R U
MW-47 U
DM-3A U/DM-3B D
A2 U
ORDINARY HIGH WATER LINE
B1B D
B1A U
LOWER SCALE PIT ROOM
SCALE PIT
EX-2 U
PZ-61 U
PZ-62 L
EX-1 U
PASSAGEWAY FROM OLD MEAL BIN
DM-4 U
B2 U
TP-4
OLD MEAL BIN
TP-2
TP-7
TP-6
W,S
WAREHOUSE
AUTOCLAVE OR VANILLIN BUILDING
DM-5 U
MW-57 U
MUSEUM OF FLIGHT PROPERTY
TRANSFORMER A EXCAVATION LOCATION
TP-8
EX-3 U
B6 U
MW-22 U
MW-20 U
PZ-60 L
IAAI LEASE PROPERTY
TP-9
MW-13 L
MW-12 U
COPPER SPILL CONTROL SUMP
1-120 SUMP
APPROXIMATE FORMER LOCATION OF OIL / WATER SEPARATOR
SUBGRADE STAIRWAY NEAR CLARIFIER
MW-42 L
MW-49 U
MW-50 L
DM-8 U
H-10 U
MW-58L
MW-55 U
MW-45 L
MW-46 L
W
W,S
DUWAMISH WATERWAY
MW-17 U
DM-7 U
MW-27 U
PZ-63 L
MW-28 U
MW-29 U
MW-53 U
MW-54 L
TP-10
MW-43 L
SLIP 6
MW-44 U
MW-41 U
MW-51 U
MW-52 L
MW-40 L
MERSIZE SUMP

EXPLANATION

- PROPERTY LINE
- APPROXIMATE LOCATION OF BARRIER WALL
- FENCE
- NORTHWEST CORNER EXCAVATION AREAS (VARYING DEPTHS)
- FORMER FACILITY STRUCTURES
- FORMER FACILITY UNDERGROUND STRUCTURES
- LOCATION OF PRE-DEMOLITION TEST PIT(S)
- W,S / W — PRE-DEMOLITION WATER AND / OR WATER AND SEDIMENT SAMPLE LOCATION
- PRE-DEMOLITION CRYSTALLINE MATERIAL SAMPLING LOCATION

MONITORING WELL NETWORK

- MONITORING WELL LOCATION
- EXTRACTION WELL LOCATION
- PIEZOMETER LOCATION
- U UPPER ZONE MONITORING POINT
- L LOWER ZONE MONITORING POINT
- D DEEP AQUIFER MONITORING POINT

HORIZONTAL DATUM
WASP NAD 83 NORTH FT

VERTICAL DATUM
MLLW FT

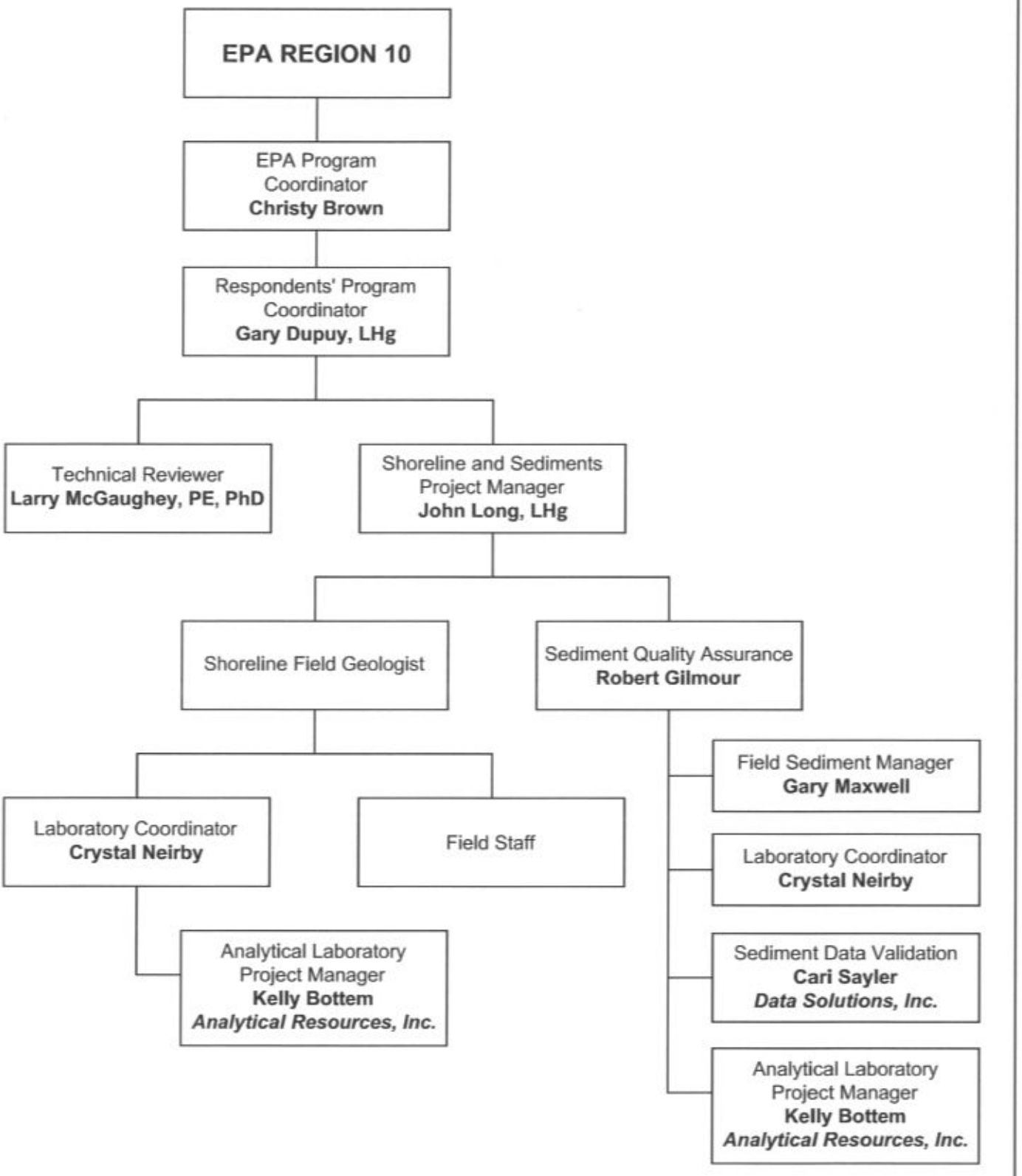
N

0 50 100
APPROXIMATE SCALE IN FEET

PRE-DEMOLITION AND REDEVELOPMENT SAMPLING LOCATION MAP
Former Rhone-Poulenc Site
Tukwila, Washington

By: APS	Date: 06/09/11	Project No. 0087690050
AMEC Geomatrix		Figure 3

Plot Date: 06/09/11 - 11:03am, Plotted by: adam.sternberg
Drawing Path: S:\8769_2006\086_FormerFeatures\, Drawing Name: FRP_FormerSumpSampLoc_060911.dwg



SEDIMENT AND SHORELINE INVESTIGATION
ORGANIZATION CHART
Former Rhone-Poulenc Site
Tukwila, Washington

By: APS	Date: 06/13/11	Project No. 0087690050
AMEC Geomatrix		Figure 4

Plot Date: 06/13/11 - 12:21pm, Plotted by: adam.stenberg
Drawing Path: S:\8769_2006\086_FormerFeatures\, Drawing Name: FRP_FormerSumpSampLoc_060911.dwg

Groundwater Pretreatment Building

ORDINARY HIGH WATER LINE

IAAI LEASE PROPERTY

MUSEUM OF FLIGHT PROPERTY

SLIP 6

AREAS WHERE BARRIER WALL WAS REINSTALLED

MW-39 L
MW-38R U
MW-42 L
DM-8 U
MW-41 U
MW-40 L
MW-45 L
MW-46 U
MW-44 U
MW-43 L
04-intsed-3
SBH-05

EXPLANATION

- DUWAMISH RIVERBANK SOIL SAMPLING LOCATION
- SLIP 6 AND SOUTHWEST CORNER SOIL AND GROUNDWATER SAMPLING LOCATION (15 TO 34 FEET IN DEPTH)
- SLIP 6 AND SOUTHWEST CORNER SOIL AND GROUNDWATER SAMPLING LOCATION (15 TO 57 FEET IN DEPTH)
- G GEOTECHNICAL SAMPLE LOCATION

OTHER FEATURES

- PROPERTY LINE
- APPROXIMATE LOCATION OF BARRIER WALL
- AREAS WHERE BARRIER WALL REINSTALLED (SEE TEXT FOR DETAILS)
- FENCE
- NORTHWEST CORNER EXCAVATION AREAS (VARYING DEPTHS)

MONITORING WELL NETWORK

- MONITORING WELL LOCATION
- EXTRACTION WELL LOCATION
- PIEZOMETER LOCATION
- U UPPER ZONE MONITORING POINT
- L LOWER ZONE MONITORING POINT
- D DEEP AQUIFER MONITORING POINT

HISTORICAL SUBSURFACE INVESTIGATION

- HISTORIC NORTHWEST CORNER SOIL SAMPLE LOCATION
- 2004 EPA INVESTIGATION SAMPLE LOCATION
- M4 HISTORIC GEOPROBE GROUNDWATER SAMPLE LOCATION
- HIGHLIGHTED ROUND 3 RCRA FACILITY INVESTIGATION SEDIMENT SAMPLING LOCATION (CH2M HILL, 1996)

HORIZONTAL DATUM
WASP NAD 83 NORTH FT

VERTICAL DATUM
MLLW FT

N

0 50 100
APPROXIMATE SCALE IN FEET

PROPOSED SHORELINE INVESTIGATION SAMPLE LOCATIONS
Former Rhone-Poulenc Site
Tukwila, Washington

By: APS	Date: 06/13/11	Project No. 0087690050
AMEC Geomatrix		Figure 5

Plot Date: 06/13/11 - 3:19pm, Plotted by: adam.stenberg
Drawing Path: S:\0769_2006\086_FormerFeatures\, Drawing Name: FRP_ShorelineProposedSamp_060211.dwg

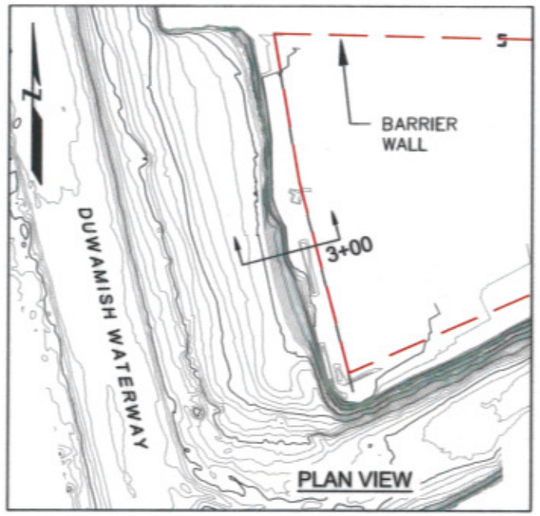
NOTE:

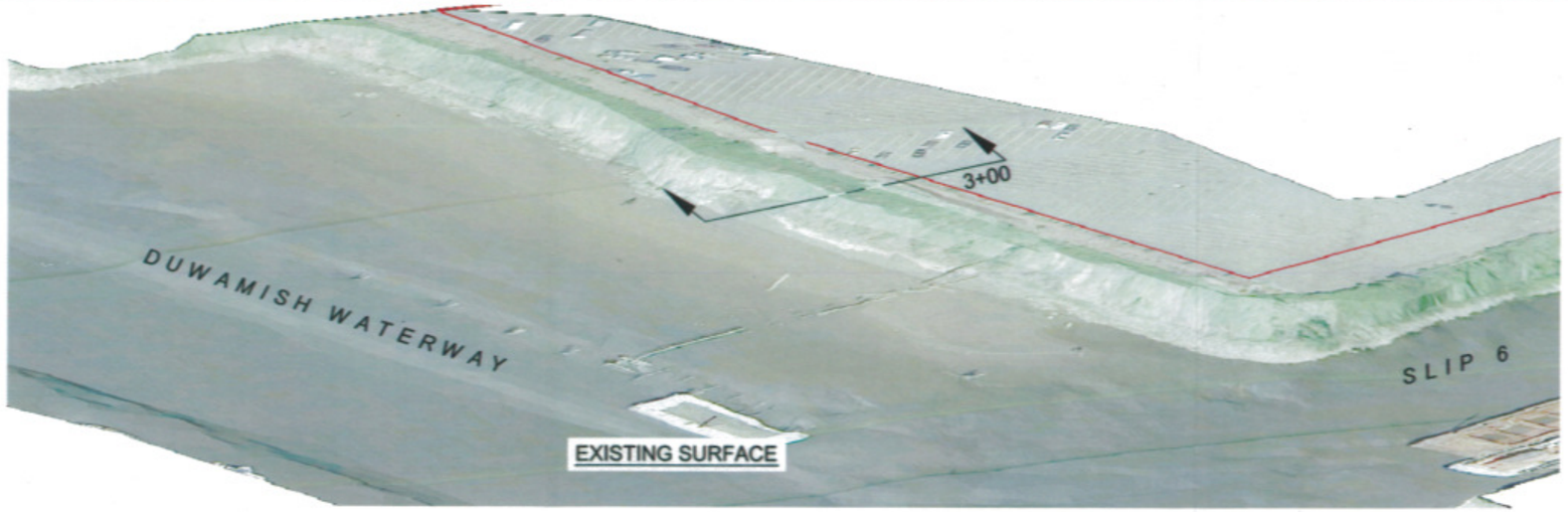
1. APPROXIMATE LOCATION OF PRIMARY COCs BASED ON FIGURE 2-2, 2-5, AND 2-11 OF THE HYDRAULIC CONTROL INTERIM MEASURES WORK PLAN (URS, 2002a)
2. STORMWATER CONVEYANCES, MANHOLES, AND CATCH BASINS BASED ON RHONE-POULENC. STORMWATER DRAINAGE PIPING DIAGRAMS FOR FACILITY ZONES 1 THROUGH 4. IDENTIFICATION OF FACILITY OUTFALLS BASED ON FIGURE 2-3 OF THE RCRA FACILITY INVESTIGATION ROUND 3 REPORT (CH2M HILL, 1996)

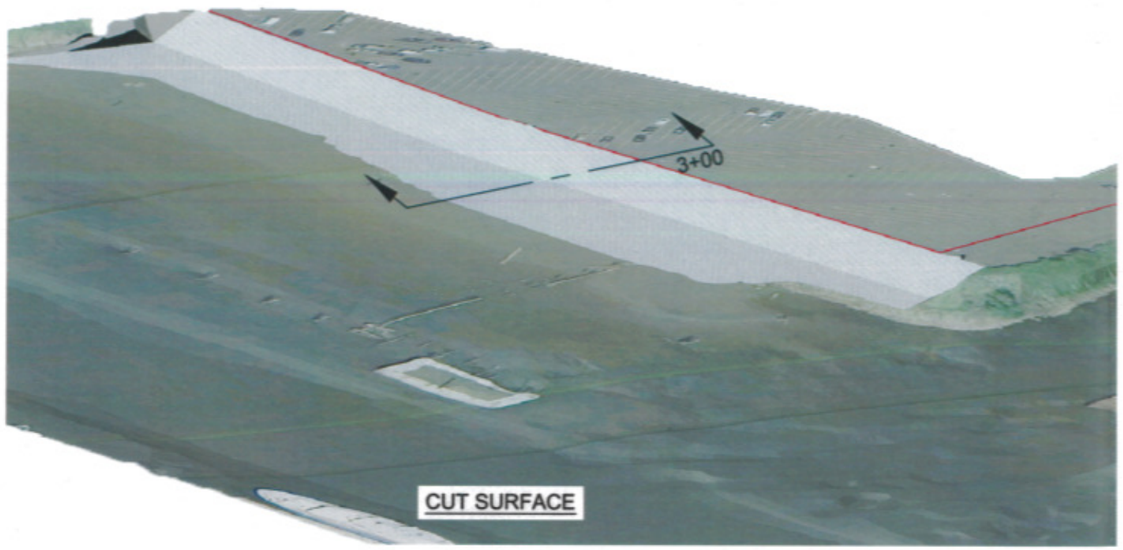
CONCEPTUAL SITE MODEL
Former Rhone-Poulenc Site
Tukwila, Washington

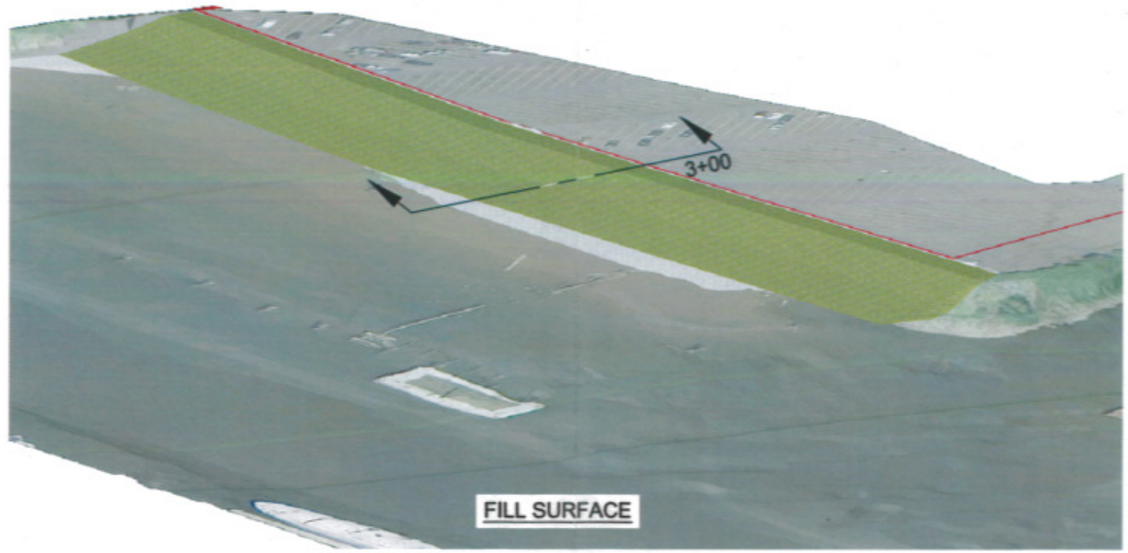
By: APS	Date: 06/13/11	Project No. 8769.005
AMEC Geomatrix		Figure 6

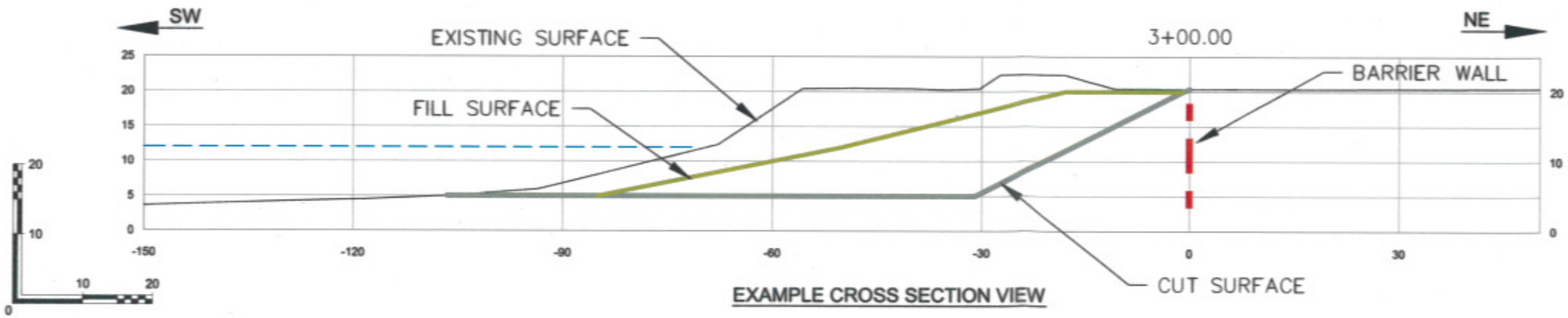
Plot Date: 06/13/11 - 3:50pm, Plotted by: adam.stenberg
Drawing Path: S:\8769_2006\086_FormerFeatures\, Drawing Name: PrimaryConceptualSiteModel_053111.dwg











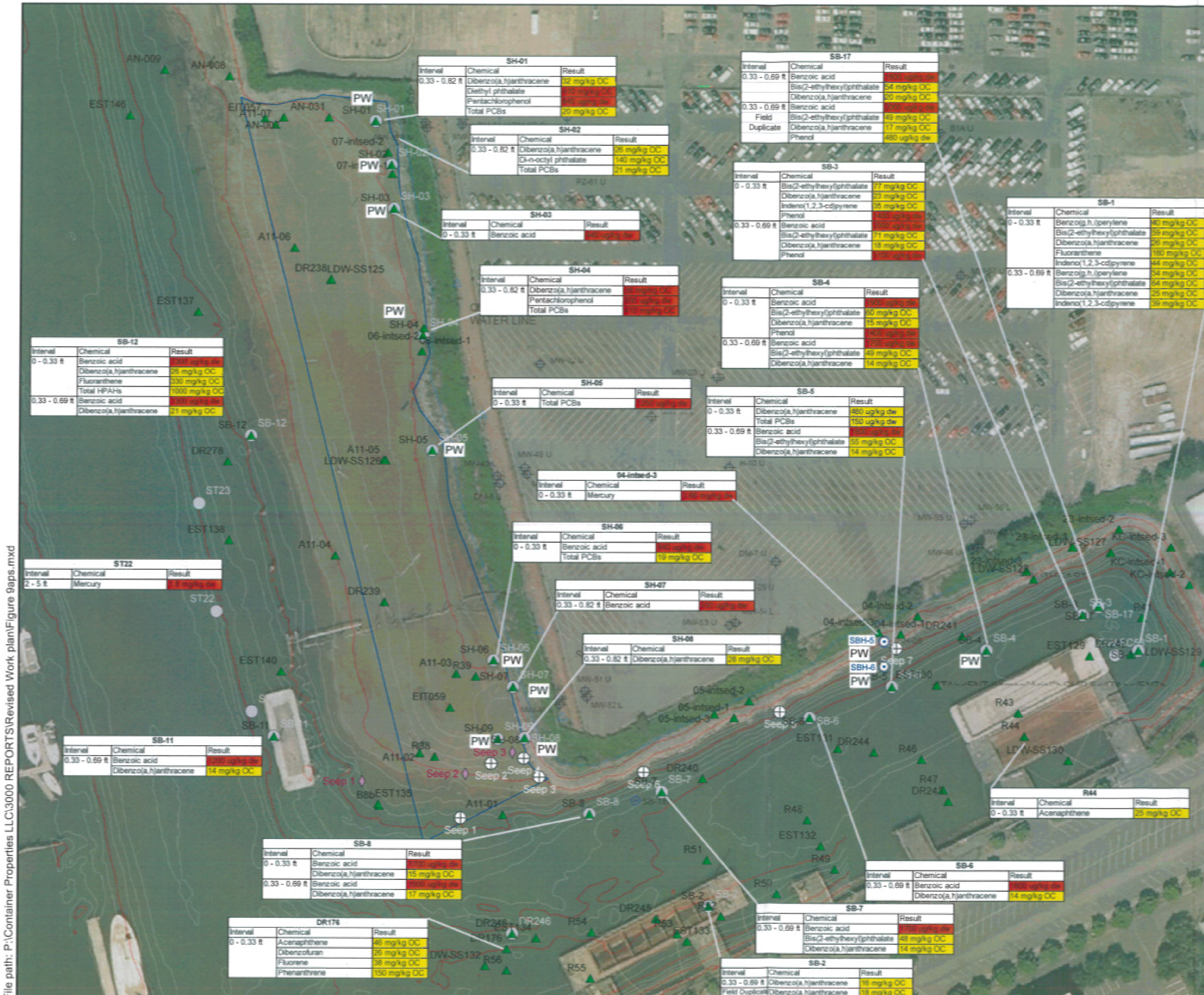
EXAMPLE CROSS SECTION VIEW

VERTICAL DATUM
MLLW FT

CONCEPTUAL HABITAT RESTORATION
Former Rhone-Poulenc Site
Tukwila, Washington

By: APS	Date: 06/13/11	Project No. 8769
AMEC Geomatrix		Figure 7

Plot Date: 06/13/11 - 12:12pm, Plotted by: adam.sternberg
Drawing Path: S:\8769_2006\087_Bank\Restoration\Rendering\CAD\, Drawing Name: WestBankVolumesHigh_060911_recover.dwg



SH-01

Interval	Chemical	Result
0.33 - 0.82 ft	Dibenzo(a,h)anthracene	32 mg/kg OC
	Diethyl phthalate	410 mg/kg OC
	Pentachlorophenol	840 ug/kg dw
	Total PCBs	20 mg/kg OC

SH-02

Interval	Chemical	Result
0.33 - 0.82 ft	Dibenzo(a,h)anthracene	26 mg/kg OC
	Di-n-octyl phthalate	140 mg/kg OC
	Total PCBs	21 mg/kg OC

SH-03

Interval	Chemical	Result
0 - 0.33 ft	Benzoic acid	840 ug/kg dw

SH-04

Interval	Chemical	Result
0.33 - 0.82 ft	Dibenzo(a,h)anthracene	36 mg/kg OC
	Pentachlorophenol	500 ug/kg dw
	Total PCBs	310 mg/kg OC

SB-12

Interval	Chemical	Result
0 - 0.33 ft	Benzoic acid	1300 ug/kg dw
	Dibenzo(a,h)anthracene	26 mg/kg OC
	Fluoranthene	330 mg/kg OC
	Total HPAHs	1000 mg/kg OC
0.33 - 0.69 ft	Benzoic acid	1300 ug/kg dw
	Dibenzo(a,h)anthracene	21 mg/kg OC

SH-05

Interval	Chemical	Result
0 - 0.33 ft	Total PCBs	1250 ug/kg dw

SB-17

Interval	Chemical	Result
0.33 - 0.69 ft	Benzoic acid	1500 ug/kg dw
	Bis(2-ethylhexyl)phthalate	54 mg/kg OC
	Dibenzo(a,h)anthracene	20 mg/kg OC
0.33 - 0.69 ft Field Duplicate	Benzoic acid	2000 ug/kg dw
	Bis(2-ethylhexyl)phthalate	49 mg/kg OC
	Dibenzo(a,h)anthracene	17 mg/kg OC
	Phenol	480 ug/kg dw

SB-3

Interval	Chemical	Result
0 - 0.33 ft	Bis(2-ethylhexyl)phthalate	77 mg/kg OC
	Dibenzo(a,h)anthracene	23 mg/kg OC
	Indeno(1,2,3-cd)pyrene	35 mg/kg OC
	Phenol	1400 ug/kg dw
0.33 - 0.69 ft	Benzoic acid	3000 ug/kg dw
	Bis(2-ethylhexyl)phthalate	71 mg/kg OC
	Dibenzo(a,h)anthracene	18 mg/kg OC
	Phenol	1100 ug/kg dw

SB-1

Interval	Chemical	Result
0 - 0.33 ft	Benzo(g,h,i)perylene	40 mg/kg OC
	Bis(2-ethylhexyl)phthalate	59 mg/kg OC
	Dibenzo(a,h)anthracene	26 mg/kg OC
	Fluoranthene	180 mg/kg OC
	Indeno(1,2,3-cd)pyrene	44 mg/kg OC
0.33 - 0.69 ft	Benzo(g,h,i)perylene	34 mg/kg OC
	Bis(2-ethylhexyl)phthalate	64 mg/kg OC
	Dibenzo(a,h)anthracene	25 mg/kg OC
	Indeno(1,2,3-cd)pyrene	39 mg/kg OC

SB-4

Interval	Chemical	Result
0 - 0.33 ft	Benzoic acid	1500 ug/kg dw
	Bis(2-ethylhexyl)phthalate	60 mg/kg OC
	Dibenzo(a,h)anthracene	15 mg/kg OC
	Phenol	1400 ug/kg dw
0.33 - 0.69 ft	Benzoic acid	1700 ug/kg dw
	Bis(2-ethylhexyl)phthalate	49 mg/kg OC
	Dibenzo(a,h)anthracene	14 mg/kg OC

SB-5

Interval	Chemical	Result
0 - 0.33 ft	Dibenzo(a,h)anthracene	460 ug/kg dw
	Total PCBs	150 ug/kg dw
0.33 - 0.69 ft	Benzoic acid	1500 ug/kg dw
	Bis(2-ethylhexyl)phthalate	55 mg/kg OC
	Dibenzo(a,h)anthracene	14 mg/kg OC

04-intsed-3

Interval	Chemical	Result
0 - 0.33 ft	Mercury	0.66 mg/kg dw

SH-06

Interval	Chemical	Result
0 - 0.33 ft	Benzoic acid	540 ug/kg dw
	Total PCBs	19 mg/kg OC

ST22

Interval	Chemical	Result
2 - 5 ft	Mercury	1.8 mg/kg dw

SH-07

Interval	Chemical	Result
0.33 - 0.82 ft	Benzoic acid	930 ug/kg dw

SH-08

Interval	Chemical	Result
0.33 - 0.82 ft	Dibenzo(a,h)anthracene	28 mg/kg OC

SB-11

Interval	Chemical	Result
0.33 - 0.69 ft	Benzoic acid	1200 ug/kg dw
	Dibenzo(a,h)anthracene	14 mg/kg OC

R44

Interval	Chemical	Result
0 - 0.33 ft	Acenaphthene	25 mg/kg OC

SB-8

Interval	Chemical	Result
0 - 0.33 ft	Benzoic acid	1700 ug/kg dw
	Dibenzo(a,h)anthracene	15 mg/kg OC
0.33 - 0.69 ft	Benzoic acid	1500 ug/kg dw
	Dibenzo(a,h)anthracene	17 mg/kg OC

SB-6

Interval	Chemical	Result
0.33 - 0.69 ft	Benzoic acid	1800 ug/kg dw
	Dibenzo(a,h)anthracene	14 mg/kg OC

SB-7

Interval	Chemical	Result
0.33 - 0.69 ft	Benzoic acid	1700 ug/kg dw
	Bis(2-ethylhexyl)phthalate	48 mg/kg OC
	Dibenzo(a,h)anthracene	14 mg/kg OC

DR176

Interval	Chemical	Result
0 - 0.33 ft	Acenaphthene	46 mg/kg OC
	Dibenzofuran	26 mg/kg OC
	Fluorene	38 mg/kg OC
	Phenanthrene	150 mg/kg OC

SB-2

Interval	Chemical	Result
0.33 - 0.69 ft	Dibenzo(a,h)anthracene	16 mg/kg OC
Field Duplicate	Dibenzo(a,h)anthracene	18 mg/kg OC

Legend

- ▲ Surface Sample Locations
- ● Subsurface Sample Locations
- ⊕ Sample Location CH2M Hill (1996)
- ◆ Sample Location Landau (1991)
- PW Pore Water Sample Location (EPA, 2005)

Chemistry Results (Detected Values) that Exceed the SMS Criteria

SB-8 — Location ID

Interval	Chemical	Result
0 - 0.33 ft	Benzoic acid	1700 ug/kg dw
	Dibenzo(a,h)anthracene	15 mg/kg OC
0.33 - 0.69 ft	Benzoic acid	1500 ug/kg dw
	Dibenzo(a,h)anthracene	17 mg/kg OC

Sample Interval

- Yellow: Result exceeds the SMS SQS but is less than the SMS CSL
- Red: Result exceeds the SMS CSL

Notes:
CSL = cleanup screening level
dw = dry weight
mg/kg = milligrams per kilogram
OC = organic carbon
SMS = Sediment Management Standards
SQS = Sediment Quality Standards
ug/kg = micrograms per kilogram

N

0 100 200 Feet

PREVIOUS SEDIMENT AND PORE WATER SAMPLE LOCATIONS AND RESULTS
Former Rhone-Poulenc Site
Tukwila, Washington

By: rhg	Date: 6/07/11	Project No. 0087690050
AMEC Geomatrix	Figure	8

File path: P:\Container Properties LLC\3000 REPORTS\Revised Work plan\Figure 9aps.mxd

Legend

- Proposed Sediment Sample Locations
- Approximate Offshore Lease Boundary
- Proposed Pore Water Sample Locations
- Highlighted Round 3 RCRA Facility Investigation Sediment Sampling Location (CH2M Hill, 1996)

PROPOSED SEDIMENT AND
PORE WATER SAMPLE LOCATIONS
Former Rhone-Poulenc Site
Tukwila, Washington

By: rhg	Date: 6/08/11	Project No. 0087690050
AMEC Geomatrix		Figure 9

File path: P:\Container Properties LLC\3000 REPORTS\Revised Work plan\Temp Figure 10aps.mxd

AMEC/Geomatrix
3500-188th St SW, Suite 600
Lynnwood,WA 98037
(425) 921-4000

CHAIN OF CUSTODY

Recorded by: _____

Checked by: _____

Place Sample ID Label Here or Write ID Number Here		Analysis Containers	Number of containers
	Date:		
	Time:		
Place Sample ID Label Here or Write ID Number Here	Date:		Number of containers
	Time:		
Place Sample ID Label Here or Write ID Number Here	Date:		Number of containers
	Time:		
Place Sample ID Label Here or Write ID Number Here	Date:		Number of containers
	Time:		
Place Sample ID Label Here or Write ID Number Here	Date:		Number of containers
	Time:		
Place Sample ID Label Here or Write ID Number Here	Date:		Number of containers
	Time:		
Place Sample ID Label Here or Write ID Number Here	Date:		Number of containers
	Time:		

Laboratory Sample Receipt

Relinquished By	Transported By	Received By
Name: Date: Time:		Name: Date: Time:
Name: Date: Time:		Name: Date: Time:
Name: Date: Time:		Name: Date: Time:

EXAMPLE CHAIN OF CUSTODY
FOR SEDIMENT SAMPLES
Former Rhone-Poulenc Site
Tukwila, Washington

By: APS	Date: 06/09/11	Project No. 0087690050
AMEC Geomatrix		Figure 11

Plot Date: 06/09/11 - 11:08am, Plotted by: adam.stenberg
Drawing Path: S:\0769_2006\086_FormerFeatures\, Drawing Name: FRP_FormerSumpSampLoc_060911.dwg

Plot Date: 06/13/11 - 12:14pm, Plotted by: adam.stenberg
Drawing Path: S:\8769_2006\086_FormerFeatures\, Drawing Name: FRP_FormerSumpSampLoc_060911.dwg

Chain of Custody Record & Laboratory Analysis Request

ARI Assigned Number:	Turn-around Requested:				Date:										
ARI Client Company:	Phone:				Page: of										
Client Contact:					No. of Coolers:	Cooler Temps:									
Client Project Name:					Analysis Requested										Notes/Comments
Client Project #:	Samplers:														
Sample ID	Date	Time	Matrix	No. Containers											
Comments/Special Instructions	Relinquished by: (Signature)		Received by: (Signature)		Relinquished by: (Signature)			Received by: (Signature)							
	Printed Name:		Printed Name:		Printed Name:			Printed Name:							
	Company:		Company:		Company:			Company:							
	Date & Time:		Date & Time:		Date & Time:			Date & Time:							

Analytical Resources, Incorporated
Analytical Chemists and Consultants
4611 South 134th Place, Suite 100
Tukwila, WA 98168
206-695-6200 206-695-6201 (fax)

Limits of Liability: *ARI will perform all requested services in accordance with appropriate methodology following ARI Standard Operating Procedures and the ARI Quality Assurance Program. This program meets standards for the industry. The total liability of ARI, its officers, agents, employees, or successors, arising out of or in connection with the requested services, shall not exceed the invoiced amount for said services. The acceptance by the client of a proposal for services by ARI release ARI from any liability in excess thereof, not withstanding any provision to the contrary in any contract, purchase order or co-signed agreement between ARI and the Client.*

Sample Retention Policy: Unless specified by workorder or contract, all water/soil samples submitted to ARI will be discarded or returned, no sooner than 90 days after receipt or 60 days after submission of hardcopy data, whichever is longer. Sediment samples submitted under PSDDA/PSEP/SMS protocol will be stored frozen for up to one year and then discarded.

EXAMPLE CHAIN OF CUSTODY
FOR SHORELINE SAMPLES
Former Rhone-Poulenc Site
Tukwila, Washington

By: APS	Date: 06/13/11	Project No. 0087690050

AMEC Geomatrix

Figure 10

APPENDIX A

APPENDIX A

Sediment Investigation Quality Assurance Project Plan

SEDIMENT INVESTIGATION
QUALITY ASSURANCE PROJECT PLAN

Former Rhone-Poulenc Site

Tukwila, Washington

Prepared for:

Container Properties, L.L.C.
Kent, Washington

Prepared by:

AMEC Geomatrix, Inc.
600 University St., Suite 1020
Seattle, Washington 98101
(206) 342-1760

June 2011

Project No. 0087690050.00005

On behalf of the respondents, I certify under penalty of law that this document and all attachments were prepared under my direction or supervision in accordance with a system designed to evaluate the information submitted. I certify that the information contained in or accompanying this Sediment Investigation Quality Assurance Project Plan, is true, accurate, and complete. As to those portions of the report for which I cannot personally verify accuracy, I certify under penalty of law that this report and all attachments were prepared in accordance with procedures designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who may manage the system, or those directly responsible for gathering the information, the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations.

By: _____ Date: June 14, 2011

Mr. Gary Dupuy, Project Coordinator

DISTRIBUTION LIST

The following individuals or entities will receive a copy of this Quality Assurance Project Plan and any subsequent revision.

EPA Project Coordinator	Christy Brown
Respondent's Project Coordinator	Gary Dupuy
AMEC Shoreline and Sediments Project Manager	John Long, AMEC Geomatrix, Inc.
AMEC Sediment QA Manager	Rob Gilmour, AMEC Geomatrix, Inc.
AMEC Field Sediment Manager	Gary Maxwell, AMEC Geomatrix, Inc.
AMEC Laboratory Coordinator	Crystal Neirby, AMEC Geomatrix, Inc.
Sediment Data Validation	Cari Sayler, Sayler Data Solutions, Inc.
Analytical Laboratory Project Manager	Kelly Bottem, Analytical Resources, Inc.

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TABLES

FIGURES

ACRONYMS & ABBREVIATIONS

°C	degrees Celsius
AMEC	AMEC Geomatrix, Inc.
ARI	Analytical Resources, Inc.
bgs	below ground surface
cm	centimeters
COC	constituent of concern
DQI	data quality indicator
DQO	data quality objective
Ecology	Washington State Department of Ecology
EDD	electronic data deliverable
EIM	Environmental Information Management
EPA	U.S. Environmental Protection Agency
LCS	laboratory control sample
MS	matrix spike
MSD	matrix-spike duplicate
NIST	National Institute of Standards and Technology
PARCC	precision, accuracy, representativeness, comparability, and completeness
PCB	polychlorinated biphenyl
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
RA/MCS	Risk Assessment/Media Cleanup Standards
RCRA	Resource Conservation and Recovery Act
RFA	RCRA Facility Assessment
RFI	RCRA Facility Investigation
RRM	Regional Reference Material
site	former Rhone-Poulenc facility located at 9229 East Marginal Way, Tukwila, Washington
SMS	Sediment Management Standards
SOP	Standard Operating Procedure
SQS	Sediment Quality Standards
SRM	Standard Reference Material
SVOCs	semivolatile organic compounds
TOC	total organic carbon
WAC	Washington Administrative Code

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SEDIMENT INVESTIGATION
QUALITY ASSURANCE PROJECT PLAN
Former Rhone-Poulenc Site
Tukwila, Washington

1.0 INTRODUCTION

This Quality Assurance Project Plan (QAPP) outlines methods, data quality objectives, and quality assurance/quality control (QA/QC) protocols for work to be conducted for the Sediments Investigation at the former Rhone-Poulenc facility (site) in Tukwila, Washington. The site is located along the Duwamish Waterway at 9229 East Marginal Way South, Tukwila, Washington. Corrective actions at the site are currently being conducted under Administrative Order on Consent No. 1091-11-20-3008(h) (Order) and are being overseen directly by the EPA. This plan conforms to the substantive requirements of the U.S. Environmental Protection Agency's (EPA's) Guidance for Quality Assurance Project Plans (EPA, 2002). The work to be conducted under this QAPP is described in the *Shoreline and Sediment Investigation Work Plan* (Work Plan; AMEC 2010a).

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2.0 PROJECT MANAGEMENT

This section describes the project management approach, including the project organization, a description of the overall project objectives and background, data quality objectives, and reporting and documentation requirements.

2.1 PROJECT ORGANIZATION

An organizational chart showing lines of authority and reporting responsibilities is presented on Figure 1.

2.1.1 Respondent's Project Coordinator

Gary Dupuy is the Respondent's Project Coordinator. His responsibilities include project direction and project oversight, site security, profiling and disposal of wastes generated, personnel access badges, space allocation, site usage, and other miscellaneous support items associated with planning and performance of the work.

2.1.2 AMEC Project Team

The consultant team working on this project consists of AMEC Geomatrix, Inc. (AMEC). AMEC is the prime consultant working under contract to Container Properties, L.L.C.

The consultant team for this project will perform the following duties:

- Communicate with and oversee the analytical laboratory to ensure that project goals are met.
- Coordinate sample analysis with the analytical laboratory.
- Provide all equipment for sediment sampling and analysis as described in the Work Plan (AMEC, 2010a).
- Establish and follow chain-of-custody procedures.
- Oversee compliance with the Site-Specific Health and Safety Plan.
- Perform field and data quality reviews.
- Prepare a data report as described in Section 4.2.

2.1.2.1 Shoreline and Sediment Investigation Project Manager

AMEC's project manager for the Shoreline and Sediment Investigation is John Long. He will be responsible for the overall supervision of the work described in this QAPP so that it meets the requirements of the overall Shoreline and Sediment Investigation.

2.1.2.2 Sediment Quality Assurance Manager

Rob Gilmour of AMEC will be the QA Manager for the sediment investigation. He will be responsible for performing field and quality reviews and verity that sampling and analysis are conducted according to the requirements specified in this QAPP.

2.1.2.3 Sediment Field Manager

Gary Maxwell will be the Field Manager for the sediment investigation. He will be responsible for:

- Verifying that all samples are collected in accordance with this QAPP;
- Establishing and following chain-of-custody procedures;
- Overseeing compliance with the Site-Specific Health and Safety Plan;
- Verifying that all sediment sampling and analysis equipment as described in the Work Plan (AMEC, 2010a) is available and in working order.

2.1.2.4 Sediment Laboratory Coordinator

Crystal Nierby of AMEC Geomatrix, Inc., will assume the role of Laboratory Coordinator for the work conducted under this QAPP. The Laboratory Coordinator will:

- Communicate with and oversee the analytical laboratory, to verify that project goals are met.
- Coordinate sample analysis with the analytical laboratory.

2.1.2.5 Sediment Data Management and Data Validation

As Laboratory Coordinator, Crystal Nierby of AMEC Geomatrix, Inc., will be responsible for data management and overall data validation. Data management will include:

- Importing the electronic data deliverable (EDD) provided by the analytical laboratory into a data management system;
- Producing analytical data tables for the data report that will be produced as part of this work;
- Producing an EDD compatible with the Washington State Department of Ecology (Ecology) Environmental Information Management (EIM) System.

Crystal Neirby will review the independent data validation conducted by Cari Sayler of Sayler Data Solutions, Inc. Cari Sayler will perform the validation of all analytical data as described in Section 5.0 of this QAPP.

2.1.2.6 *Analytical Laboratory Project Manager*

Analytical testing will be conducted by Analytical Resources, Inc. (ARI), of Tukwila, Washington. ARI is a Washington-accredited full-service chemical analytical laboratory. Kelly Bottem will be the ARI Project Manager. ARI will perform the following duties:

- Perform all laboratory chemical analyses.
- Meet data quality requirements outlined in Section 2.3 of this QAPP.
- Provide storage for all frozen archived sediment samples in a temperature-monitored freezer at -18 degrees Celsius (°C).

2.2 PROJECT DESCRIPTION

The former Rhone-Poulenc facility is located along the Duwamish Waterway at 9229 East Marginal Way South, Tukwila, Washington (Figure 2). Corrective actions at the site are currently being conducted under Administrative Order on Consent No. 1091-11-20-3008(h) (Order) and are being overseen directly by the EPA.

Since site closure in 1991, extensive investigations have been completed at the site to evaluate environmental impacts to soil and groundwater from the former vanillin manufacturing plant. The investigations have followed the Resource Conservation and Recovery Act (RCRA) process from an initial RCRA Facility Assessment (RFA) (PRC, 1990) through a 1991 Site Assessment (Landau, 1991), the RCRA Facility Investigation (RFI) (CH2M HILL, 1995), and a Risk Assessment/Media Cleanup Standards Report (RA/MCS) (AGI Technologies, 1999). Studies completed subsequent to the RFI include geoprobe and geotechnical investigations conducted in support of interim measure design (URS, 2002a) and a geoprobe investigation (AGI Technologies, 2001).

The primary constituents of concern (COCs) for the site are:

- Toluene, an industrial solvent used in the vanillin manufacturing process;
- Copper in soils and groundwater resulting from vanillin black liquor solids used for weed control, various releases of contaminated surface runoff waters and process waste waters, and strainer solids from vanillin manufacture; and
- Groundwater affected by elevated pH due to caustic releases.

Toluene-affected groundwater is limited primarily to the southwest portion of the site. Copper-affected groundwater and groundwater having elevated pH due to the caustic release are limited to the western side and southwestern corner of the site, based on historical data. Other metals are present to a limited extent in groundwater. Other COCs for the site include polycyclic aromatic hydrocarbons (PAHs), methylene chloride, benzene, arsenic, chromium, lead, mercury, nickel, and vanadium. In

addition, semivolatile organic compounds (SVOCs), including pentachlorophenol, have been documented at the site.

Elevated concentrations of polychlorinated biphenyls (PCBs) have also been observed in an area affected by past releases from a former PCB-containing compressor. PCB-contaminated soils around the compressor pad and a decommissioned underground drain line were removed during two separate interim measures (Rhodia, 1998; Geomatrix 2006a). Sources of metals (such as the use of metals sludge for weed control or the burial of autoclave solids) and other contaminants are described in the RFI report (CH2M HILL, 1995).

The interim groundwater remedy used at this site is hydraulic containment. A hydraulic control interim measure (HCIM) was constructed at the site from January through July 2003, consistent with the EPA-approved work plan (URS, 2002b). The HCIM consists of a low-permeability, subsurface barrier wall with a groundwater extraction and treatment system designed to maintain an inward-directed groundwater gradient. The extracted groundwater is treated using granular activated carbon (GAC) and discharged to a publicly owned treatment works (POTW).

In 2006, the entire facility underwent redevelopment, and additional subsurface investigations were performed. The property was split into two parcels, the East Parcel and the West Parcel. The East Parcel was extensively investigated and remediated. EPA provided a partial determination of "Corrective Action Complete without Controls" for the East Parcel in a letter dated December 20, 2006 (EPA, 2006). The partial determination was made since a portion of the property, approximately 2,000 square feet in size in the extreme southwestern corner of the East Parcel, was found to have soil and groundwater impacted with toluene above project-specific cleanup goals. Corrective actions were undertaken for this portion of the property using combinations of air sparge, biovent, and/or soil vapor extraction systems to treat toluene in the soil and groundwater. Some combination of these systems was operated from December 2008 until June 2010, when the systems were shut down. Container Properties is continuing to monitor groundwater quality in this area to confirm that corrective actions are complete. The East Parcel is now owned by the Museum of Flight, and throughout this work plan the former East Parcel will be referred to as the Museum of Flight Property.

The West Parcel was regraded and repaved as part of redevelopment activities. The West Parcel is now leased by Container Properties to International Auto Auctions, Inc. (IAAI). This work plan applies to investigation activities associated with the former West Parcel, which will be referred to in this work plan as the IAAI Lease Property, the former Rhone-Poulenc facility, or the site.

The HCIM has been in operation at the site since August 2003, and continues controlling migration of groundwater within the barrier wall. However, the HCIM was not designed to control or capture

groundwater remaining outside the barrier wall. Just prior to redevelopment, limited excavation of soils affected by copper and petroleum hydrocarbons was completed in the northwest corner of the IAAI lease property in an area just outside the barrier wall (see Figure 2 for location). Soil sampling was conducted in the northwest corner, but the nature and extent of copper- and petroleum-affected soil was not determined to the south of northwest corner sampling location 42.

In a letter dated April 28, 2009, EPA requested that additional investigation be completed in three areas of the former Rhone-Poulenc facility: the Slip 6 bank, the western riverbank, and the sediments in the offshore area (EPA, 2009b). The Respondents, including Container Properties, requested a meeting with EPA to negotiate options regarding completion of the additional work. Due to several scheduling conflicts, this meeting was held at EPA Region 10 offices on August 12, 2010.

EPA subsequently sent a letter to the Respondents dated August 18, 2010, indicating that the additional work EPA requested is still required and that the Respondents should submit a work plan for the additional work by October 18, 2010 (EPA, 2010b). The Respondents requested an extension to the October 18, 2010, deadline for submittal of the work plan (AMEC, 2010). In its reply dated September 16, 2010, EPA approved the Respondents' request for an extension of the deadline for submittal of the work plan to November 19, 2010 (EPA, 2010c). This QAPP covers the work that will be conducted to delineate the nature and extent of the contaminated sediments in the tidal areas of the IAAI Lease Property.

2.3 QUALITY OBJECTIVES AND CRITERIA FOR ANALYTICAL DATA

Analytical data should meet project data quality objectives (DQOs). The DQOs were developed so that analytical data are accurate enough to compare with the Washington State Sediment Quality Standards (SQS) for marine sediments (Table 1). The SQS for a majority of the organic compounds and for polychlorinated biphenyls (PCBs) are based on carbon-normalized concentrations. Therefore, total organic carbon (TOC) content of the samples must also be analyzed. Comparison of carbon-normalized values against the SQS listed in Table 1 may be inappropriate if TOC values are below 0.5 percent or above 4 percent. At TOC concentrations below 0.5 percent and above 4 percent, the project DQOs for data related to organic compounds and PCBs must be accurate at the dry-weight-based standards in Table 1. No SQS criteria have been established for vanadium or dieldrin; therefore, the DQOs for these analytes are based on the analytical laboratory reporting limits.

To meet the goal of returning data accurate within the SQS or other criteria, data quality indicators (DQIs) for specific measured parameters, including the familiar PARCC parameters (precision, accuracy, representativeness, comparability, and completeness), and for sensitivity are required. The basis for assessing each of these elements of data quality is discussed in the following sections.

Quality control (QC) limits for precision and accuracy are identified in Tables 2 through 7 for each method and matrix.

2.3.1 Precision

Precision measures the reproducibility of measurements. It is strictly defined as the degree of mutual agreement among independent measurements as the result of repeated application of the same process under similar conditions. Analytical precision is the measurement of the variability associated with duplicate (two) or replicate (more than two) analyses. If the recoveries of analytes in the laboratory control sample (LCS) are within established control limits, then precision is within limits. Total precision is the measurement of the variability associated with the entire sampling and analysis process and is determined by analysis of duplicate or replicate field samples. Total precision measures the variability introduced by both the laboratory and field operations. Field-duplicate samples (10 percent frequency) and matrix-duplicate spiked samples (1 per 20 samples) will be analyzed to assess field and analytical precision using the relative percent difference between the duplicate sample results. For replicate analyses, the relative standard deviation is determined.

2.3.2 Accuracy

Accuracy is a statistical measurement of correctness and includes components of random error (variability due to imprecision) and systematic error. It therefore reflects the total error associated with a measurement. A measurement is accurate when the value reported does not differ from the true value or known concentration of the spike or standard. Analytical accuracy is measured by comparing the percent recovery of analytes spiked into an LCS to a control limit. For compounds such as PCBs, surrogate compound recoveries are also used to assess accuracy and method performance for each sample analyzed.

Both accuracy and precision are calculated for each analytical batch, and the associated sample results are interpreted by considering these specific measurements. The sample batch for both the LCS and method blank includes up to 20 samples extracted together. The formula for calculation of accuracy returns a percent recovery from pure and sample matrices. Limits of accuracy for the various analytical methods are contained in Tables 2 through 7.

2.3.3 Representativeness

Objectives for representativeness are defined for each sampling and analysis task and are a function of investigative objectives. Representativeness will be achieved by using the standard field, sampling, and analytical procedures. Representativeness is also determined by appropriate program design, with consideration of elements such as proper sampling locations, sampling procedures, and sampling intervals. Decisions regarding sample locations and sample intervals are documented in Section 3.0.

2.3.4 Comparability

Comparability is the confidence with which one data set can be compared to another data set. An objective for this QA/QC program is to produce data comparable to previously collected data. The range of field conditions encountered is considered in determining comparability. Comparability will be achieved by using standard methods for sampling and analysis, reporting data in standard units, using Regional Reference Material (RRM) or Standard Reference Materials (SRM), and using standard reporting formats. Field documentation using standardized data-collection forms will support the assessment of comparability.

2.3.5 Completeness

Completeness is calculated and reported for each method, matrix, and analyte combination. The number of valid results divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness of the data set. For completeness requirements, valid results are all results not qualified with an "R" flag (see Table 8 for an explanation of data flagging criteria). The requirement for completeness for this project is 90 percent for the sediment samples scheduled for the initial round of analyses.

2.4 Documentation and Records

Data and log forms produced in the field will be reviewed daily by the person recording the data so that any errors or omissions can be corrected. All completed data sheets are removed from the field clipboard and photocopied; the original data sheets are filed in a fireproof file cabinet and the photocopies stored in the project file. All data transcribed from field forms into electronic forms and tables will be 100 percent verified for accuracy and freedom from transcription errors.

Laboratory documentation will consist of a case narrative, providing descriptions of any problems and corrective actions, copies of the chain-of-custody forms, tabulated analytical results, data qualifiers, and blank and matrix-spike results with calculated percent recoveries and differences. A more detailed documentation package (raw data, analyst's reports, extraction logs, chromatograms, etc.) will be provided by the laboratory in case the basic data review discussed in Section 5.1 encounters deficiencies requiring more thorough laboratory documentation.

Field documentation will consist of forms presented in the Work Plan (AMEC 2010a). All project documentation records for the sediment investigation will be copied. Original documentation records will be kept on file at the offices of AMEC in Lynnwood, Washington. Copies of all field documentation will be kept in the master project file maintained by AMEC's Seattle office. All data generated as part of this QAPP will be retained at the offices of AMEC

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3.0 DATA GENERATION AND ACQUISITION

This section describes the methods and protocols to be used for sample collection and analysis, including laboratory analytical methods and quality control procedures.

3.1 SAMPLING PROCESS DESIGN

The primary objective of the sampling design is to assess the nature and extent of potential surface and subsurface contamination in the tideflat and offshore areas that can be attributed to potential sources in the upland portions of the site. The results from this sediment investigation and the previous sampling efforts will be reviewed for trends in concentration that indicate offshore transport of onshore sources of contamination. EPA has identified the constituents of concern for the sediments at the project site as the Washington State SQS list of chemicals (WAC 173-204-320), as well as the pesticide dieldrin and the metal vanadium.

A majority of the proposed sample locations are laid out in a systematic, triangular grid arrangement within the property boundaries, as described in the Work Plan (AMEC, 2010a). In response to comments made by EPA, additional judgmental samples will be included in the sediment investigation. Additional grab and core samples will be collected to investigate sediment quality near a groundwater seep off the southwest corner of the site (near the mouth of Slip 6). A surface sample will also be collected as close as possible to a previous intertidal sample location with elevated mercury results on the north bank of Slip 6 (previous location 04-intsed-3; proposed sample location RP-19; Figure 2). Hand collection methods will be used to collect sediment at sample location RP-19.

Detailed core-collection and processing procedures are presented in the Work Plan (AMEC, 2010a). A summary of the sampling methods is presented below.

3.2 SEDIMENT SAMPLING METHODS

Surface sediment samples will be collected using a grab sampler to collect the top 0.33 foot (10 centimeters [cm]) of sediment. Samples to characterize the subsurface sediments will be collected using an impact corer.

The impact corer uses the impact from the linear pneumatic hammer to drive a 4-inch-square aluminum core into the sediment. The impact corer allows for a continuous core sample to be collected over the depth that the tube is driven. Paired penetration and recovery measurements are collected during driving to develop a recovery curve for each core. The bottom of each core tube will be fitted with a hinged core catcher to prevent loss of the sediment during extraction.

The intent of the core sampling is to determine the depth below mudline where the sediment concentrations of COCs are below the SQS. Impact cores will be advanced 15 feet below mudline or until refusal. Full core penetration and sample recovery may not be possible at all locations using the proposed sampling equipment. Native sediments can be very dense, and recovery of deeper sample intervals becomes difficult with greater penetration. If penetration is less than the proposed target depth, then the recovered cores will be evaluated to determine their acceptability using the following protocol:

- If the contact with native sediments is present in the core and samples can be collected below the contact, and the core was driven to refusal using the available equipment, additional sampling attempts at that location may not be required.
- If penetration is less than the target depth and the native contact is not visible in the core or if samples cannot be collected below the contact, the corer will be relocated a minimum of 6 feet (2 meters) from the original location, and a second core will be attempted.

If a second core is attempted, penetration is less than the target depth, and no native contact is visible or samples cannot be collected below the contact, then no further sampling using the impact corer will be attempted at that location, and AMEC's project manager and the EPA project coordinator will be notified. If deeper sediment samples are needed at a location to characterize the sediments, then an additional round of sampling using other equipment may be required.

Detailed sediment collection and handling procedures are presented in the Work Plan (AMEC, 2010a). The handling and processing of sediment cores will occur within a secured exclusion zone using Level D personal protective equipment (PPE) following the requirements specified in the Site-Specific Health and Safety Plan.

A single grab will be collected and processed at a time. A single core tube will be handled and processed at a time. Cores collected with the impact corer will be held for a maximum of 24 hours before processing. Unprocessed cores held more than 8 hours will be chilled with ice.

Table 9 lists sample locations, sample intervals, and proposed initial analyses for the sediment sampling. A total of 20 locations plus 2 duplicate locations will be sampled as part of this investigation. Samples will be assigned a unique identifier using a sample prefix of RP for former Rhone-Poulenc site. Stations will be identified by a sequential number appended to the sample prefix.

At all grab sample locations, sediment from the surface to a depth of 0.33 foot (10 cm) will be collected. At all core sampling locations, discrete samples will be collected from each core at 1-foot in situ depth intervals starting at the intervals identified in Table 9 to the proposed target depth of 15 feet below ground surface (bgs) or refusal.

If the volume of recovered sediment within a depth interval is insufficient to perform all the analyses identified in Table 9, additional sediment from the next deeper interval will be added to provide sufficient volume. The next sample will be collected from the next complete and intact 1-foot in situ depth interval. The sampling routine may be modified in the field based on site conditions at the direction of the field geologist.

At stations where multiple cores are collected, the impact core with the best penetration and recovery will be processed. Samples will be collected to the deepest usable sample interval.

3.3 SAMPLE HANDLING AND CUSTODY

This section describes sampling handling procedures and field QC protocols.

3.3.1 Sediment Analysis Schedule

The COCs for the sediment investigation are the Washington Sediment Management Standards (SMS) SQS list of constituents of concern, plus vanadium and dieldrin. Table 9 provides a list of the sediment samples proposed to be analyzed initially. All of the samples in this group of samples will initially be analyzed for TOC, the SMS list of COCs, dieldrin, and vanadium. The surface grab samples will also be analyzed for grain size. Additional samples may be analyzed based on sediment characteristics observed by the field geologist during core processing. Surplus sample volume from analyzed samples will be frozen (-18°C) and archived at the analytical laboratory. All samples collected but not initially analyzed will also be frozen and archived at the analytical laboratory.

3.3.2 Additional Sediment Analyses

The results of the initial round of testing will be reviewed and, based on the results of the initial round of analyses additional samples may be analyzed to refine the depth of sediment containing elevated levels of COCs. Additional analyses may target specific COCs.

3.3.3 Field Quality Control

The field QC protocol will include collection and analysis of duplicate samples at approximately a 10 percent frequency. Field QC samples for the sediment investigation will be collected at RP-04 and RP-10; these duplicate locations will be designated RP-21 and RP-22 respectively. Duplicate grabs collected at RP-04 and RP-10 will be analyzed for the same analytes as the parent samples. Samples from four depth intervals (2 to 3 feet, 4 to 5 feet, 8 to 9 feet, and 12 to 13 feet) will be analyzed from the duplicate core collected at location RP-04 (RP-20); three sample intervals (2 to 3 feet, 4 to 5 feet, and 8 to 9 feet) will be analyzed from the duplicate core collected at location RP-10 (RP-21). Additional QC samples may be analyzed to obtain an approximate 10 percent frequency.

Decontamination (rinsate) blanks will not be collected during the grab sampling portion of the investigation. Sediment touching the sides of the grab sampler will not be collected. All sample collection and homogenation will be performed using precleaned sampling equipment.

Decontamination (rinsate) blanks will not be collected during the impact coring portion of the investigation. All core sampling will be conducted using precleaned sampling equipment (see the Work Plan; AMEC 2010).

Samples will be handled using standard chain-of-custody procedures. Data and log forms produced in the field will be reviewed daily by the person recording the data, so that any errors or omissions can be corrected. All completed data sheets are removed daily from the field clipboard and photocopied; the original data sheets are filed in a fireproof file cabinet and the photocopies stored in the project file. All data transcribed from field forms into electronic forms and tables will be 100 percent verified for accuracy and freedom from transcription errors.

3.4 ANALYTICAL METHODS

The analysis methods chosen for the sediment samples must be able to return accurate results at the concentrations listed in Table 1. The SMS chemical criteria for a majority of the organic compounds in Table 1 (nonionizable organic compounds) are expressed on an organic carbon normalized basis in milligrams per kilogram (mg/kg) carbon. The use of an organic carbon-normalized chemical criterion may be inappropriate for sediment with TOC values less than 0.5 percent or greater than 4.0 percent. Comparison to the lowest apparent effects threshold (LAET) dry weight equivalent (in micrograms per kilogram [µg/kg]) of the SMS SQS criteria (Table 1) may be appropriate if the TOC is less than 0.5 percent or greater than 4 percent. The test method selected to achieve these results is described in Table 10 along with the laboratory reporting limits for the analysis provided by ARI. Laboratory reporting limits for metals are expressed in mg/kg. Laboratory reporting limits for organic compounds are expressed in µg/kg. The standard reporting levels for the test methods will achieve the DQOs provided the quantitation limits are not elevated due to dilution. If the reporting limit of a compound is above the appropriate SQS criterion but the compound is not detected, additional cleanup or analysis methods may be used to achieve lower reporting limits. Detected results below the laboratory reporting limit but above the method detection limit will be assigned a J (estimated value) qualifier by the laboratory. If a compound has a reporting limit above the SMS chemical criterion, then the compound will be considered to exceed the relevant criterion.

Because the core sediment samples will not be homogenized in the field (see the Work Plan; AMEC 2010a), ARI will homogenize the entire core sediment sample before sample aliquots are removed for analysis of metals, SVOCs, PCBs, dieldrin, and TOC.

As described in the SMS, total PCB concentrations will be calculated by summing the detected concentrations for nine Aroclors (Aroclor 1016, 1221, 1232, 1242, 1248, 1254, 1260, 1262, and 1268). Undetected Aroclors will not be included in the calculation of total PCB values. If all nine Aroclors are reported as undetected, the highest undetected value is reported as the total PCB value. In the event that Aroclor concentrations are extremely elevated and therefore require dilution, a different extraction procedure may be used.

Elevated reporting limits for certain Aroclors can result from several causes, most commonly a result of dilution or sample interferences. In very-high-concentration samples, dilution may be necessary to accurately quantitate the Aroclor(s) present at the highest concentration(s). This can result in elevated quantitation limits for other Aroclor(s) as reporting limits must be multiplied by the dilution factor. Sample interferences can raise the baseline of the chromatogram above reporting-limit resolution. If such interferences result in elevated reporting limits above DQOs, ARI will perform additional cleanup of the extract to try to remove these interferences (see Section 4.1, Assessments and Response Actions).

3.5 QUALITY CONTROL

Field QC checks include collection and analysis of duplicate samples (10 percent frequency) and standardized sampling documentation forms (see Work Plan; AMEC 2010a). Decontamination (rinsate) blanks will not be collected during grab sampling since sediment that touches the sides of the sampler will not be collected and precleaned sampling and processing equipment will be used. Decontamination blanks will not be collected during the core sampling because only precleaned, sampling equipment will be used (Work Plan; AMEC 2010). Trip blanks will not be analyzed.

Laboratory QC checks include use of standard EPA analytical methods, analysis of method-specified QC samples (such as analysis method blanks, spikes, and surrogates), and meeting method-specified calibration and system performance criteria. These QC criteria are detailed in Tables 2 through 7. Analyses will be carried out under the laboratory's Standard Operating Procedures (SOPs).

A suitable RRM for PCBs and an SRM for TOC will be run with every third batch of samples, beginning with the first batch. One matrix spike/matrix-spike duplicate (MS/MSD) will be run with each batch (each batch with less than 20 samples) to evaluate matrix interferences and recoveries. Additional sample volume will be collected to meet the analysis needs.

3.6 Instrument/Equipment Testing, Inspection, Maintenance, and Calibration

3.6.1 Laboratory Equipment

Analytical instruments will be calibrated according to the analytical methods specified in the laboratory SOPs. All analytes reported will be present in the initial and continuing calibrations, and these calibrations will meet the acceptance criteria specified in Tables 2 through 7. Records of standard preparation and instrument calibration will be maintained, and calibration standards shall be traceable to standard materials.

Instrument calibration will be checked at the frequency specified in Tables 2 through 7 for the corresponding analytical method using materials prepared independently of the LCSs. Multipoint calibrations will contain the minimum number of calibration points specified in the method, with all points used for the calibration being contiguous. If more than the minimum number of standards are analyzed for the initial calibration, all of the standards analyzed will be included in the initial calibration. The continuing calibration verification cannot be used as the LCS.

3.7 Inspection and Acceptance of Supplies and Consumables

The Field Manager will be responsible for ensuring that all supplies necessary to conduct the sampling, including collecting, processing, and transporting samples, are available and in good working order at the beginning of the fieldwork. The Field Manager will monitor supplies and equipment throughout sampling and replenish or replace as necessary.

3.8 Nondirect Measurements

No nondirect measurements will be made on this project.

3.9 Data Management

The analytical and field data will be compiled into an EIM-compatible electronic data deliverable for submission to EPA. The analytical data will also be maintained in ARI's electronic Laboratory Information Management System.

4.0 ASSESSMENT AND OVERSIGHT

This section presents the protocol for conducting field and laboratory assessments and specifies reporting requirement for this project.

4.1 ASSESSMENTS AND RESPONSE ACTIONS

4.1.1 Field

The Field Manager will be responsible for correcting equipment malfunctions during the sediment sampling. In addition to equipment failures, conditions that require a modification of the intent of the sampling program will be coordinated with EPA by the Field Manager or the Respondent's Project Coordinator. All response actions will be documented on field forms or in a logbook.

4.1.2 Analytical Laboratory

ARI participates in Ecology's Environmental Laboratory Accreditation Program and has participated in the EPA Contract Laboratory Program. The laboratory is periodically audited by a variety of outside agencies, including EPA, Ecology, the U.S. Army Corps of Engineers, and the Washington State Department of Health. Results of recent audits are available from ARI.

Corrective actions will occur whenever the QC limits for one of the methods specified in Tables 2 through 7 are exceeded. Details of the corrective actions are specified in the laboratory SOPs for each analytical method.

Whenever a corrective action occurs, the Laboratory Manager is notified. If the corrective action is judged to be routine, such as a slight exceedance of a percent-recovery limit, the corrective action will be implemented without notification of the Shoreline and Sediment Project Manager. If the corrective action requires reanalysis or reextraction, the Shoreline and Sediment Project Manager and Laboratory Coordinator will be notified.

For PCB analyses, if environmental interferences result in ARI not attaining the target reporting limit, the Shoreline and Sediment Project Manager and Laboratory Coordinator will be notified and gel-permeation cleanup (EPA Method 3640A) will be performed before reanalysis. Because sediment samples will be frozen following removal of material for the initial analyses, which allows for a 6-month hold time, the laboratory will be able to re-extract and reanalyze samples well within the required holding-time interval.

4.2 REPORTING

A data report summarizing the results of the characterization will be prepared by AMEC and the Respondent's Project Coordinator for submittal to EPA. This report will include a narrative of field

activities, chain-of-custody records, a Level 3 data review, data tables and maps for sample locations, data tables and maps summarizing the results of the analytical analyses, and electronic data tables.

5.0 DATA VALIDATION

This section describes procedures for data validation, verification, and usability.

5.1 DATA REVIEW, VERIFICATION, AND VALIDATION

All data packages will be verified and validated following a Level 2B evaluation (EPA, 2009a) (also referred to as a QA1 review by Ecology) (Ecology, 2008). The verification and validation includes the following summarized steps:

- Verify that the lab utilized the specified extract, analysis, and cleanup methods.
- Review sample holding time.
- Verify that sample numbers and analyses match those requested on the chain-of-custody form.
- Verify that the required reporting limits have been achieved.
- Verify that field duplicates, matrix spikes, and lab control samples were run at the proper frequency and have met QC criteria.
- Verify that the surrogate compound analyses have been performed and have met QC criteria.
- Verify that initial and continuing calibrations were run at the proper frequency and have met acceptance criteria.
- Verify that the lab blanks are free of contaminants.

Data review will be performed in accordance with EPA's functional guidelines for data validation (EPA, 2008; EPA, 2010a), the analytical method referenced by the laboratory, AMEC data review procedures, and the laboratory quality control limits. EPA (2008, 2010a) guidance is written specifically for the Contract Laboratory Program and will be modified for the purposes of this review where procedures differ from EPA SW-846 method requirements. Following the data review, data qualifiers assigned by the laboratory may be amended using the data validation qualifiers described in Table 8.

5.2 RECONCILIATION WITH USER REQUIREMENTS

Following receipt of all of the analytical data reports, the Shoreline and Sediment Project Manager and the project team will review the sample results to determine if they fall within the acceptance limits and goals described in this Quality Assurance Project Plan. If the DQOs do not meet project requirements, the data may be discarded and reanalysis performed. This decision will be made jointly between the project team and the Respondent's Project Coordinator. EPA will be informed of any

decisions to reanalyze. If the failure is traced to the analytical laboratory, sample handling, extraction, or instrument calibration and maintenance, techniques will be reassessed before reanalysis.

6.0 HEALTH AND SAFETY

Worker health-and-safety requirements will be provided in a Site-Specific Health and Safety Plan to be prepared for sediment investigation in accordance with applicable state regulations for hazardous-waste-site workers (Washington Administrative Code [WAC] 296-843).

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7.0 REFERENCES

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TABLES

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TABLE 1

DATA QUALITY OBJECTIVES FOR THE CONSTITUENTS OF CONCERN
PRACTICAL QUANTITATION REQUIREMENTS

Former Rhone-Poulenc Site
Tukwila, Washington

Chemical Parameter	Management Standards [1]		LAET [4]	Sediment DQOs [5]
	SQS [2]	CSL [3]		
Metals	**mg/kg dry wt**	**mg/kg dry wt**	**mg/kg dry wt**	**mg/kg dry wt**
Arsenic	57	93	57	—
Cadmium	5.1	6.7	5.1	—
Chromium	260	270	260	—
Copper	390	390	390	—
Lead	450	530	450	—
Mercury	0.41	0.59	0.41	—
Silver	6.1	6.1	6.1	—
Vanadium	—	—	—	0.3
Zinc	410	960	410	—
Nonionizable Organic Compounds	**mg/kg carbon**	**mg/kg carbon**	**µg/kg dry wt**	**µg/kg dry wt**
Aromatic Hydrocarbons				
Total LPAH	370	780	5,200	—
Naphthalene	99	170	2,100	—
Acenaphthylene	66	66	1,300	—
Acenaphthene	16	57	500	—
Fluorene	23	79	540	—
Phenanthrene	100	480	1,500	—
Anthracene	220	1,200	960	—
2-Methylnaphthalene	38	780	670	—
Total HPAH	960	5,300	12,000	—
Fluoranthene	160	1,200	1,700	—
Pyrene	1,000	1,400	2,600	—
Benz[a]anthracene	110	270	1,300	—
Chrysene	110	460	1,400	—
Total benzofluoranthenes	230	450	3,200	—
Benzo[a]pyrene	99	210	1,600	—
Indeno[1,2,3-c,d]pyrene	34	88	600	—
Dibenzo[a,h]anthracene	12	33	230	—
Benzo[g,h,i]perylene	31	78	670	—
Chlorinated Benzenes				
1,2-Dichlorobenzene	2.3	2.3	35	—
1,4-Dichlorobenzene	3.1	9	110	—
1,2,4-Trichlorobenzene	0.81	1.8	31	—
Hexachlorobenzene	0.38	2.3	22	—
Phthalate Esters				
Dimethyl phthalate	53	53	71	—
Diethyl phthalate	61	110	200	—
Di-n-butyl phthalate	220	1,700	1,400	—
Butyl benzyl phthalate	4.9	64	63	—
Bis[2-ethylhexyl] phthalate	47	78	1,300	—
Di-n-octyl phthalate	58	4,500	6,200	—

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TABLE 1

DATA QUALITY OBJECTIVES FOR THE CONSTITUENTS OF CONCERN
PRACTICAL QUANTITATION REQUIREMENTS

Former Rhone-Poulenc Site
Tukwila, Washington

Chemical Parameter	Management Standards [1]		LAET [4]	Sediment DQOs [5]
	SQS [2]	CSL [3]		
Miscellaneous				
Dibenzofuran	15	58	540	—
Hexachlorobutadiene	3.9	6.2	11	—
N-nitrosodiphenylamine	11	11	28	—
Total PCBs	12	65	130	—
Ionizable Organic Compounds	**µg/kg dry wt**	**µg/kg dry wt**	**µg/kg dry wt**	**µg/kg dry wt**
Phenol	420	1,200	420	—
2-Methylphenol	63	63	63	—
4-Methylphenol	670	670	670	—
2,4-Dimethylphenol	29	29	29	—
Pentachlorophenol	360	690	360	—
Benzyl alcohol	57	73	57	—
Benzoic acid	650	650	650	—
Pesticides	**µg/kg dry wt**	**µg/kg dry wt**	**µg/kg dry wt**	**µg/kg dry wt**
Dieldrin	—	—	—	2

Notes

1. SMS criteria for nonionizable organic compounds are expressed in mg/kg carbon, since SMS for most nonionizable organic compounds are generally expressed as a carbon-normalized value. However, values for nonionizable organic compounds are usually not carbon-normalized in sediments with TOC values above 4% or below 0.5%, and the so dry-weight equivalent (LAET) values for these constituents are also provided. In these cases, the dry weight equivalent values are generally used instead.
2. SQS = Sediment Quality Standards (WAC 173-204-320).
3. CSL = Cleanup Screening Levels (WAC 173-204-520).
4. LAET = Lowest Apparent Effects Threshold. Dry weight equivalent of the SMS "SQS."
5. Analytical laboratory reporting limits.

Abbreviations

µg/kg = micrograms per kilogram
CSL = cleanup screening level
DQOs = data quality objectives
dry wt = dry weight
HPAH = high-molecular-weight aromatic hydrocarbons
LPAH = low-molecular-weight aromatic hydrocarbons
mg/kg = milligrams per kilogram
PAH = polycyclic aromatic hydrocarbons
PCBs = polychlorinated biphenyls
SQS = Sediment Quality Standards
TOC = total organic carbon
WAC = Washington Administrative Code
wt = weight

TABLE 2

SUMMARY OF QUALITY OBJECTIVES FOR METHOD 8082—PCBs

Former Rhone-Poulenc Site
Tukwila, Washington

Quality-Control Element	Frequency of Implementation	Acceptance Criteria
Initial Calibration	After CCVs fail	RSD ≤ 20% or r ≥ 0.995
Continuing Calibration Verification (CCV)	At the beginning and end of analytical sequence, and every 12 hours	% Recovery = 75% to 125%
Method Blank (MB)	1 every 20 samples; minimum of 1 per extraction batch	Analytes < RL
Laboratory Control Sample (LCS)	1 every 20 samples; minimum of 1 per extraction batch	Solids: % Recovery = 37% to 116%
Matrix Spike (MS)	1 per 20 samples	% Recovery = 37% to 116%
Matrix Duplicate (MD) or Matrix-Spike Duplicate (MSD)	1 per 20 samples	RPD ≤ 50%
Regional Reference Material (RRM)	1 per 50 samples	Advisory Limits: Average +/- 2SD % Recovery 19% to 112%
Surrogates	Every sample as specified	% Recovery = 34% to 141%
Target Analyte Confirmation	Every detected compound	RPD ≤ 40%

Abbreviations
PCBs = polychlorinated biphenyls
RL = reporting limit
RPD = relative percent difference
RSD = relative standard deviation
SD = standard deviation

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TABLE 3

SUMMARY OF QUALITY OBJECTIVES FOR METHOD 8270D—SVOCs

Former Rhone-Poulenc Site
Tukwila, Washington

Quality-Control Element	Frequency of Implementation	Acceptance Criteria
Initial Calibration	After CCV fails	r > 0.990 or RSD < 20%, RRF >0.050 for SPCC and >0.010 for other cmpds.
Continuing Calibration Verification (CCV)	At the beginning of each 12 hour shift	%D < 20% for CCC and < 40% for other cmpds, RRF >0.050 for SPCC and >0.010 for other cmpds.
Method Blank (MB)	1 every 20 samples; minimum of 1 per extraction batch	Analytes < RL
Laboratory Control Sample (LCS)	1 every 20 samples; minimum of 1 per extraction batch	Solids: % Recovery = 10% to 160% B/N cmpds % Recovery = 10% to 140% A cmpds
Matrix Spike (MS)	1 per 20 samples	Solids: % Recovery = 10% to 160% B/N cmpds % Recovery = 10% to 140% A cmpds
Matrix Duplicate (MD) or Matrix Spike Duplicate (MSD)	1 per 20 samples	RPD < 50%
Surrogates:	Every sample as specified	
Interference-Free Matrix		Interference-Free Matrix Solids: % Recovery = 34% to 106% B/N cmpds % Recovery = 14% to 109% A cmpds
Project Sample Matrix		Project Sample Matrix % Recovery = 30% to 113% A cmpds % Recovery = 10% to 116% A cmpds

Abbreviations

%D = percent difference
A = acid compounds (cmpds).
B/N = base, neutral compounds (cmpds).
CCC = calibration check compounds
cmpds = compounds
RL = reporting limit
RPD = relative percent difference
RRF = relative response factor
RSD = relative standard deviation
SPCC = system performance check compounds.
SVOCs = semivolatile organic compounds

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TABLE 4

SUMMARY OF QUALITY OBJECTIVES FOR METHOD 6010/200.8—ICP and ICPMS METALS

Former Rhone-Poulenc Site
Tukwila, Washington

Quality-Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria
Initial Calibration	Option 1: 1 standard and 1 blank, and a low-level-check standard at RL	Daily	Option 1: Low-level-check standard ± 1 RL
	Option 2: 3 standards and 1 blank		Option 2: $r > 0.995$
Instrumental Precision	% RSD 3 integrations (exposures)	Each calibration and calibration verification standards (ICV/CCV)	% RSD < 5%
Initial Calibration Verification (ICV)	Midlevel (2nd source) verification	After initial calibration	% Recovery 90% to 110%
Initial Calibration Blank (ICB)	Interference-Free Matrix to assess analysis contamination	After initial calibration	Analytes < RL
Continuing Calibration Verification (CCV)	Midlevel verification	Every 10 samples and at end of analytical sequence	% Recovery 90% to 110%
Continuing Calibration Blank (CCB)	Interference-Free Matrix to assess analysis contamination	Every 10 samples and at end of analytical sequence	Analytes < RL
Method Blank (MB)	Interference-Free Matrix to assess overall method contamination	1 every 20 samples; minimum of 1 per extraction batch	Analytes < RL or < 1/10th lowest sample instrument concentration.
Laboratory Control Sample (LCS)	Interference-Free Matrix containing all target analytes	1 every 20 samples; minimum of 1 per extraction batch	% Recovery = 80% to 120%
			Sporadic Marginal Failures[1]:
			% Recovery = 80% to 140%
Matrix Spike (MS)	Sample matrix spiked with all or a subset of target analytes prior to digestion	1 per 20 samples	% Recovery = 75% to 125%
Matrix Duplicate (MD) or Matrix Spike Duplicate (MSD)	Refer to text for MD or MS	1 per 20 samples	RPD < 20%

Notes

1. The number of sporadic marginal failure (SMF) allowances depends on the number of target analytes reported from the analysis. In the instance of only seven metals, one SMF is allowed.

Abbreviations
RL = reporting limit
RPD = relative percent difference
RSD = relative standard deviation
std = standard

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TABLE 5

SUMMARY OF QUALITY OBJECTIVES FOR METHOD 7000 SERIES—CVAA METALS

Former Rhone-Poulenc Site
Tukwila, Washington

Quality-Control Element	Description of Element	Frequency of Implementation	Acceptance Criteria
Initial Calibration	3 standards and 1 blank	Daily	r > 0.995
Instrumental Precision	RPD of 2 injections	All standards, and ICV/CCV	RPD < 10%
Initial Calibration Verification (ICV)	Midlevel (2nd source) verification	After initial calibration	% Recovery = 90% to 110%
Initial Calibration Blank (ICB)	Interference-free matrix to assess analysis contamination	After initial calibration	Analytes < RL
Continuing Calibration Blank (CCB)	Interference-free matrix to assess analysis contamination	Every 10 samples and at end of analytical sequence	Analytes < RL
Continuing Calibration Verification (CCV)	Midlevel verification	Every 10 samples and at end of analytical sequence	% Recovery = 80% to 120%
Method Blank (MB)	Interference-free matrix to assess overall method contamination	1 every 20 samples; minimum of 1 per extraction batch	Analytes < RL
Laboratory Control Sample (LCS)	Interference-free matrix containing target analytes	1 every 20 samples; minimum of 1 per extraction batch	% Recovery = 80% to 120%
Matrix Spike (MS)	Sample matrix spiked with target analytes prior to digestion	1 per 20 samples	% Recovery = 75% to 125%
Matrix Duplicate (MD) or Matrix-Spike Duplicate (MSD)	Refer to text for MD or MS	1 per 20 samples	RPD <20%
Post-Digestion Spike (PDS)	Sample digestate spiked with target analytes	As needed to confirm matrix effects	% Recovery = 85% to 115%

Abbreviations
RL = reporting limit
RPD = relative percent difference

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TABLE 6

SUMMARY OF QUALITY OBJECTIVES FOR METHOD 8260C—SEDIMENT VOCs

Former Rhone-Poulenc Site
Tukwila, Washington

Quality Control Element	Frequency of Implementation	Acceptance Criteria [1]
Initial Calibration	As needed	RSD ≤ 20%, $r^2 \geq 0.990$
Initial Calibration Verification (ICV)	After initial calibration	% Recovery = 70 - 130%
Continuing Calibration Verification (CCV)	Every 12 hours	% Drift ≤ 20%, %D ≤ 20%
Method Blank (MB)	1 every 20 samples; minimum of 1 per extraction batch	Analytes < RL
Laboratory Control Sample (LCS)	1 every 20 samples; minimum of 1 per extraction batch	Soil: % Recovery [2] = Vinyl Chloride: 63 - 137% Low Lev *Cis*-1, 2-Dichloroethene: 80 - 120% Trichloroethene: 80 - 120% Other analytes and Med Level: various%
Matrix Spike (MS)	1 per 20 samples	% Recovery [2] = Vinyl chloride: 63 - 137% *cis*-1, 2-Dichloroethene: 80 - 120% Trichloroethene: 80 - 120% Other analytes: various%
Matrix-Spike Duplicate (MSD)	1 per 20 samples	RPD ≤ 30%
Surrogates: Interference-Free Matrix Project Sample Matrix	Every sample as specified	 Interference-free matrix Soil: % Recovery = 79% to 121% Project sample matrix % Recovery = 30% to 160%
Target Analyte Confirmation Duplicate	Every detected compound	RPD ≤ 30%

Notes

1. Control limits, reporting limits, and method detection limits are subject to change based on annual verification and review by the analytical laboratory.
2. Control limits based on a 5 mL purge volume.

Abbreviations

%D = percent difference
Lev = level
Med = medium
RL = reporting limit
RPD = relative percent difference
RSD = relative standard deviation

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TABLE 7

SUMMARY OF QUALITY OBJECTIVES FOR METHOD 8081A—PESTICIDES

Former Rhone-Poulenc Site
Tukwila, Washington

Quality Control Element	Frequency of Implementation	Acceptance Criteria [1]
Initial Calibration	As needed	RSD ≤ 20%
Initial Calibration Verification (ICV)	After initial calibration	% Recovery = 80-120%
Continuing Calibration Verification (CCV)	Every 12 hours	% Drift ≤ 20%, %D ≤ 20%
Method Blank (MB)	1 every 20 samples; minimum of 1 per extraction batch	Analytes < RL
Laboratory Control Sample (LCS)	1 every 20 samples; minimum of 1 per extraction batch	Soil: % Recovery = 37% to 150%
Matrix Spike (MS)	1 per 20 samples	% Recovery = 37% to 150%
Matrix-Spike Duplicate (MSD)	1 per 20 samples	RPD ≤ 50%
Surrogates: Interference-Free Matrix Project Sample Matrix	Every sample as specified	 Interference-free matrix Soil: % Recovery = 53% to 113% Project sample matrix % Recovery = 26% to 143%
Target Analyte Confirmation Duplicate	Every detected compound	RPD ≤ 40%

Notes

1. Control limits, reporting limits, and method detection limits are subject to change based on annual verification and review by the analytical laboratory.

Abbreviations

%D = percent difference
RL = reporting limit
RPD = relative percent difference
RSD = relative standard deviation

TABLE 8

DATA QUALIFIERS

Former Rhone-Poulenc Site
Tukwila, Washington

Qualifier	Description
U	The compound was analyzed for, but not detected.
UJ	The compound was analyzed for, but was not detected; the associated quantitation limit is an estimate because quality-control criteria were not met.
J	The analyte was positively identified, but the associated numerical value is an estimated quantity because quality-control criteria were not met or because concentrations reported are less than the quantitation limit or lowest calibration standard.
N	The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification.
NJ	The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
R	Quality control indicates that data are unusable (compound may or may not be present). Reanalysis is necessary for verification.
UY	PCB methods only. The laboratory uses the Y qualifier when interferences (usually the presence of the overlapping PCB Aroclor at high concentrations) cause the detection limit to be raised. The Y-flagged Aroclor may be present at concentrations at or below the limit reported, but in the opinion of the analyst, insufficient information is present to confirm the detection according to the method's protocols. The concentration should be treated as a non-detected value at a raised detection limit. The "U" has been added to the lab's "Y" qualifier to stress that the sample should be treated as a non-detected value.

Abbreviations
PCB = polychlorinated biphenyl

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TABLE 9

SEDIMENT SAMPLE LOCATIONS AND PROPOSED INITIAL ANALYSIS SCHEDULE

Former Rhone-Polenc Site
Tukwila, Washington

Proposed Sample Location	State Plane Coordinates (WA SPC North NAD 83; Survey Feet)		Estimated Mudline Elevation (feet MLLW) [1]	Sample Type	Samples Collected [2]	Preliminary List of Initial Samples Analyzed [3]	Analyses
	Easting	Northing					
RP-01	1276587	193530	5.3	Grab	top 10 cm	top 10 cm	SMS SQS, V, dieldrin, TS, TOC, grain size
RP-01	1276587	193530	5.3	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, V, dieldrin, TS, TOC
						4- to 5-foot interval	SMS SQS, V, dieldrin, TS, TOC
						8- to 9-foot interval	SMS SQS, V, dieldrin, TS, TOC
						12- to 13-foot interval	SMS SQS, V, dieldrin, TS, TOC
RP-02	1276518	193463	2.7	Grab	top 10 cm	top 10 cm	SMS SQS, V, dieldrin, TS, TOC, grain size
RP-02	1276518	193463	2.7	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, V, dieldrin, TS, TOC
						4- to 5-foot interval	SMS SQS, V, dieldrin, TS, TOC
						8- to 9-foot interval	SMS SQS, V, dieldrin, TS, TOC
						12- to 13-foot interval	SMS SQS, V, dieldrin, TS, TOC
RP-03	1276611	193436	4.2	Grab	top 10 cm	top 10 cm	SMS SQS, V, dieldrin, TS, TOC, grain size
RP-03	1276611	193436	4.2	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, V, dieldrin, TS, TOC
						4- to 5-foot interval	SMS SQS, V, dieldrin, TS, TOC
						8- to 9-foot interval	SMS SQS, V, dieldrin, TS, TOC
						12- to 13-foot interval	SMS SQS, V, dieldrin, TS, TOC
RP-04 [4]	1276541	193369	2.7	Grab	top 10 cm	top 10 cm	SMS SQS, V, dieldrin, TS, TOC, grain size
RP-04 [4]	1276541	193369	2.7	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, V, dieldrin, TS, TOC
						4- to 5-foot interval	SMS SQS, V, dieldrin, TS, TOC
						8- to 9-foot interval	SMS SQS, V, dieldrin, TS, TOC
						12- to 13-foot interval	SMS SQS, V, dieldrin, TS, TOC
RP-20	1276541	193369	2.7	Grab	top 10 cm	top 10 cm	SMS SQS, V, dieldrin, TS, TOC, grain size
RP-20	1276541	193369	2.7	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, V, dieldrin, TS, TOC
						4- to 5-foot interval	SMS SQS, V, dieldrin, TS, TOC
						8- to 9-foot interval	SMS SQS, V, dieldrin, TS, TOC
						12- to 13-foot interval	SMS SQS, V, dieldrin, TS, TOC
RP-05	1276634	193342	4.3	Grab	top 10 cm	top 10 cm	SMS SQS, V, dieldrin, TS, TOC, grain size
RP-05	1276634	193342	4.3	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, V, dieldrin, TS, TOC
						4- to 5-foot interval	SMS SQS, V, dieldrin, TS, TOC
						8- to 9-foot interval	SMS SQS, V, dieldrin, TS, TOC
						12- to 13-foot interval	SMS SQS, V, dieldrin, TS, TOC
RP-06	1276565	193275	2.8	Grab	top 10 cm	top 10 cm	SMS SQS, V, dieldrin, TS, TOC, grain size
RP-06	1276565	193275	2.8	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, V, dieldrin, TS, TOC
						4- to 5-foot interval	SMS SQS, V, dieldrin, TS, TOC
						8- to 9-foot interval	SMS SQS, V, dieldrin, TS, TOC
						12- to 13-foot interval	SMS SQS, V, dieldrin, TS, TOC
RP-07	1276657	193249	4.7	Grab	top 10 cm	top 10 cm	SMS SQS, V, dieldrin, TS, TOC, grain size
RP-07	1276657	193249	4.7	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, V, dieldrin, TS, TOC
						4- to 5-foot interval	SMS SQS, V, dieldrin, TS, TOC
						8- to 9-foot interval	SMS SQS, V, dieldrin, TS, TOC
						12- to 13-foot interval	SMS SQS, V, dieldrin, TS, TOC
RP-08	1276588	193182	2.7	Grab	top 10 cm	top 10 cm	SMS SQS, V, dieldrin, TS, TOC, grain size

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TABLE 9

SEDIMENT SAMPLE LOCATIONS AND PROPOSED INITIAL ANALYSIS SCHEDULE

Former Rhone-Polenc Site
Tukwila, Washington

Proposed Sample Location	State Plane Coordinates (WA SPC North NAD 83; Survey Feet)		Estimated Mudline Elevation (feet MLLW) [1]	Sample Type	Samples Collected [2]	Preliminary List of Initial Samples Analyzed [3]	Analyses
	Easting	Northing					
RP-08	1276588	193182	2.7	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, V, dieldrin, TS, TOC
						4- to 5-foot interval	SMS SQS, V, dieldrin, TS, TOC
						8- to 9-foot interval	SMS SQS, V, dieldrin, TS, TOC
						12- to 13-foot interval	SMS SQS, V, dieldrin, TS, TOC
RP-09	1276681	193155	5.6	Grab	top 10 cm	top 10 cm	SMS SQS, V, dieldrin, TS, TOC, grain size
RP-09	1276681	193155	5.6	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, V, dieldrin, TS, TOC
						4- to 5-foot interval	SMS SQS, V, dieldrin, TS, TOC
						8- to 9-foot interval	SMS SQS, V, dieldrin, TS, TOC
						12- to 13-foot interval	SMS SQS, V, dieldrin, TS, TOC
RP-10 [5]	1276611	193088	2.5	Grab	top 10 cm	top 10 cm	SMS SQS, V, dieldrin, TS, TOC, grain size
RP-10 [5]	1276611	193088	2.5	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, V, dieldrin, TS, TOC
						4- to 5-foot interval	SMS SQS, V, dieldrin, TS, TOC
						8- to 9-foot interval	SMS SQS, V, dieldrin, TS, TOC
						12- to 13-foot interval	SMS SQS, V, dieldrin, TS, TOC
RP-21	1276611	193088	2.5	Grab	top 10 cm	top 10 cm	SMS SQS, V, dieldrin, TS, TOC, grain size
RP-21	1276611	193088	2.5	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, V, dieldrin, TS, TOC
						4- to 5-foot interval	SMS SQS, V, dieldrin, TS, TOC
						8- to 9-foot interval	SMS SQS, V, dieldrin, TS, TOC
						(12- to 13-foot sample duplicate not required)	
RP-11	1276704	193061	5.8	Grab	top 10 cm	top 10 cm	SMS SQS, V, dieldrin, TS, TOC, grain size
RP-11	1276704	193061	5.8	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, V, dieldrin, TS, TOC
						4- to 5-foot interval	SMS SQS, V, dieldrin, TS, TOC
						8- to 9-foot interval	SMS SQS, V, dieldrin, TS, TOC
						12- to 13-foot interval	SMS SQS, V, dieldrin, TS, TOC
RP-12	1276635	192994	2.2	Grab	top 10 cm	top 10 cm	SMS SQS, V, dieldrin, TS, TOC, grain size
RP-12	1276635	192994	2.2	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, V, dieldrin, TS, TOC
						4- to 5-foot interval	SMS SQS, V, dieldrin, TS, TOC
						8- to 9-foot interval	SMS SQS, V, dieldrin, TS, TOC
						12- to 13-foot interval	SMS SQS, V, dieldrin, TS, TOC
RP-13	1276728	192968	5.6	Grab	top 10 cm	top 10 cm	SMS SQS, V, dieldrin, TS, TOC, grain size
RP-13	1276728	192968	5.6	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, V, dieldrin, TS, TOC
						4- to 5-foot interval	SMS SQS, V, dieldrin, TS, TOC
						8- to 9-foot interval	SMS SQS, V, dieldrin, TS, TOC
						12- to 13-foot interval	SMS SQS, V, dieldrin, TS, TOC
RP-14	1276658	192900	2.4	Grab	top 10 cm	top 10 cm	SMS SQS, V, dieldrin, TS, TOC, grain size
RP-14	1276658	192900	2.4	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, V, dieldrin, TS, TOC
						4- to 5-foot interval	SMS SQS, V, dieldrin, TS, TOC
						8- to 9-foot interval	SMS SQS, V, dieldrin, TS, TOC
						12- to 13-foot interval	SMS SQS, V, dieldrin, TS, TOC
RP-15	1276751	192874	3.4	Grab	top 10 cm	top 10 cm	SMS SQS, V, dieldrin, TS, TOC, grain size

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TABLE 9

SEDIMENT SAMPLE LOCATIONS AND PROPOSED INITIAL ANALYSIS SCHEDULE

Former Rhone-Polenc Site
Tukwila, Washington

Proposed Sample Location	State Plane Coordinates (WA SPC North NAD 83; Survey Feet)		Estimated Mudline Elevation (feet MLLW) [1]	Sample Type	Samples Collected [2]	Preliminary List of Initial Samples Analyzed [3]	Analyses
	Easting	Northing					
RP-15	1276751	192874	3.4	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, V, dieldrin, TS, TOC
						4- to 5-foot interval	SMS SQS, V, dieldrin, TS, TOC
						8- to 9-foot interval	SMS SQS, V, dieldrin, TS, TOC
						12- to 13-foot interval	SMS SQS, V, dieldrin, TS, TOC
RP-16	1276681	192807	0.4	Grab	top 10 cm	top 10 cm	SMS SQS, V, dieldrin, TS, TOC, grain size
RP-16	1276681	192807	0.4	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, V, dieldrin, TS, TOC
						4- to 5-foot interval	SMS SQS, V, dieldrin, TS, TOC
						8- to 9-foot interval	SMS SQS, V, dieldrin, TS, TOC
						12- to 13-foot interval	SMS SQS, V, dieldrin, TS, TOC
RP-17	1276771	192784	-0.4	Grab	top 10 cm	top 10 cm	SMS SQS, V, dieldrin, TS, TOC, grain size
RP-17	1276771	192784	-0.4	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, V, dieldrin, TS, TOC
						4- to 5-foot interval	SMS SQS, V, dieldrin, TS, TOC
						8- to 9-foot interval	SMS SQS, V, dieldrin, TS, TOC
						12- to 13-foot interval	SMS SQS, V, dieldrin, TS, TOC
RP-18	1276750	192820	4.0	Grab	top 10 cm	top 10 cm	SMS SQS, V, dieldrin, TS, TOC, grain size
RP-18	1276750	192820	4.0	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, V, dieldrin, TS, TOC
						4- to 5-foot interval	SMS SQS, V, dieldrin, TS, TOC
						8- to 9-foot interval	SMS SQS, V, dieldrin, TS, TOC
						12- to 13-foot interval	SMS SQS, V, dieldrin, TS, TOC
RP-19	1277194	192953	6.0	Hand	top 10 cm	top 10 cm	Mercury
RP-20	1276907	192761	-3.5	Core	1-foot intervals from surface to up to 15 feet below mudline	2- to 3-foot interval	SMS SQS, V, dieldrin, TS, TOC
						4- to 5-foot interval	SMS SQS, V, dieldrin, TS, TOC
						8- to 9-foot interval	SMS SQS, V, dieldrin, TS, TOC
						12- to 13-foot interval	SMS SQS, V, dieldrin, TS, TOC

Notes

1. Estimated from bathymetric survey.
2. See Section 3.4.1.1 of the Work Plan (AMEC, 2010a) for discussion of the target sample depth.
3. Intervals to be analyzed may be changed based on sediment characteristics observed by the field geologist during core processing and actual recovery depth.
4. Duplicate core collected at this location (sample designated RP-20).
5. Duplicate core collected at this location (sample designated RP-21).

Abbreviation

MLLW = mean lower low water
NAD = North American Datum
SMS = Sediment Management Standards
SQS = Sediment Quality Standards
TS = Total Solids
V = vanadium
WA SPC = Washington State Plane Coordinates

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TABLE 10

ANALYTICAL METHODOLOGIES AND REPORTING LIMITS

Former Rhone-Poulenc Site
Tukwila, Washington

Analyte	Sample Preparation/ Extraction	Analytical Method	Detection Limit	Reporting Limit [1, 2]
Conventionals				
Total Organic Carbon (Sediment)	ARI 602S	EPA 9060/Plumb 1981	—	200 mg/kg
Total Solids	ARI 639S	EPA 160.1/PSEP	—	0.10%
Metals (mg/kg)				
Arsenic	EPA 3050	EPA 200.8 (ICPMS)	0.068	0.5
Cadmium	EPA 3050	EPA 6010 (ICP-OES)	0.11	0.2
Chromium	EPA 3050	EPA 6010 (ICP-OES)	0.27	0.5
Copper	EPA 3050	EPA 6010 (ICP-OES)	0.05	0.2
Lead	EPA 3050	EPA 6010 (ICP-OES)	0.13	2.0
Mercury	EPA 7471A	EPA 7471A (CVAA)	0.0013	0.05
Silver	EPA 3050	EPA 6010 (ICP-OES)	0.03	1.0
Vanadium	EPA 3050	EPA 6010 (ICP-OES)	0.06	0.3
Zinc	EPA 3050	EPA 6010 (ICP-OES)	0.12	1.0
Nonionizable Organic Compounds				
Aromatic Hydrocarbons (µg/kg)				
Total LPAH	—	—	—	—
Naphthalene	EPA 3550B	EPA 8270D - PSEP	2.71	20
Acenaphthylene	EPA 3550B	EPA 8270D - PSEP	3.00	20
Acenaphthene	EPA 3550B	EPA 8270D - PSEP	3.30	20
Fluorene	EPA 3550B	EPA 8270D - PSEP	3.57	20
Phenanthrene	EPA 3550B	EPA 8270D - PSEP	3.61	20
Anthracene	EPA 3550B	EPA 8270D - PSEP	4.37	20
2-Methylnaphthalene	EPA 3550B	EPA 8270D - PSEP	2.99	20
Total HPAH	—	—	—	—
Fluoranthene	EPA 3550B	EPA 8270D - PSEP	4.38	20
Pyrene	EPA 3550B	EPA 8270D - PSEP	4.78	20
Benz[a]anthracene	EPA 3550B	EPA 8270D - PSEP	4.62	20
Chrysene	EPA 3550B	EPA 8270D - PSEP	5.82	20
Total benzofluoranthenes	EPA 3550B	EPA 8270D - PSEP	5.70	40
Benzo[a]pyrene	EPA 3550B	EPA 8270D - PSEP	5.13	20
Indeno[1,2,3-c,d]pyrene	EPA 3550B	EPA 8270D - PSEP	5.05	20
Dibenzo[a,h]anthracene	EPA 3550B	EPA 8270D - PSEP	4.54	20
Benzo[g,h,i]perylene	EPA 3550B	EPA 8270D - PSEP	4.76	20
Chlorinated Benzenes (µg/kg)				
1,2-Dichlorobenzene	EPA 3550B	EPA 8270D - PSEP	2.96	20
1,4-Dichlorobenzene	EPA 3550B	EPA 8270D - PSEP	2.73	20
1,2,4-Trichlorobenzene	EPA 3550B	EPA 8260C - PSEP	3.79	5
Hexachlorobenzene	EPA 3550B	EPA 8081A - PSEP	3.38	1

TABLE 10

ANALYTICAL METHODOLOGIES AND REPORTING LIMITS

Former Rhone-Poulenc Site
Tukwila, Washington

Analyte	Sample Preparation/ Extraction	Analytical Method	Detection Limit	Reporting Limit [1, 2]
Phthalate Esters (µg/kg)				
Dimethyl phthalate	EPA 3550B	EPA 8270D - PSEP	3.72	20
Diethyl phthalate	EPA 3550B	EPA 8270D - PSEP	3.75	20
Di-n-butyl phthalate	EPA 3550B	EPA 8270D - PSEP	4.68	20
Butyl benzyl phthalate	EPA 3550B	EPA 8270D - PSEP	4.11	20
Bis[2-ethylhexyl] phthalate	EPA 3550B	EPA 8270D - PSEP	8.73	20
Di-n-octyl phthalate	EPA 3550B	EPA 8270D - PSEP	5.22	20
Miscellaneous (µg/kg)				
Dibenzofuran	EPA 3550B	EPA 8270D - PSEP	3.15	20
Hexachlorobutadiene	EPA 3550B	EPA 8081A - PSEP	0.138	0.5
N-Nitrosodiphenylamine	EPA 3550B	EPA 8270D - PSEP	12.8	20
Total PCBs	PSDDA Sonication [3] (low levels)	EPA 8082	9.33 to 10.82	20 µg/kg per Aroclor
Ionizable Organic Compounds (µg/kg)				
Phenol	EPA 3550B	EPA 8270D - PSEP	3.80	20
2-Methylphenol	EPA 3550B	EPA 8270D - PSEP	5.34	20
4-Methylphenol	EPA 3550B	EPA 8270D - PSEP	4.82	20
2,4-Dimethylphenol	EPA 3550B	EPA 8270D - PSEP	7.98	20
Pentachlorophenol	EPA 3550B	EPA 8270D - PSEP	27.4	100
Benzyl alcohol	EPA 3550B	EPA 8270D - PSEP	46.1	100
Benzoic acid	EPA 3550B	EPA 8270D - PSEP	42.6	200
Pesticides (µg/kg)				
Dieldrin	EPA 3550B	EPA 8081A - PSEP	0.100	1.0

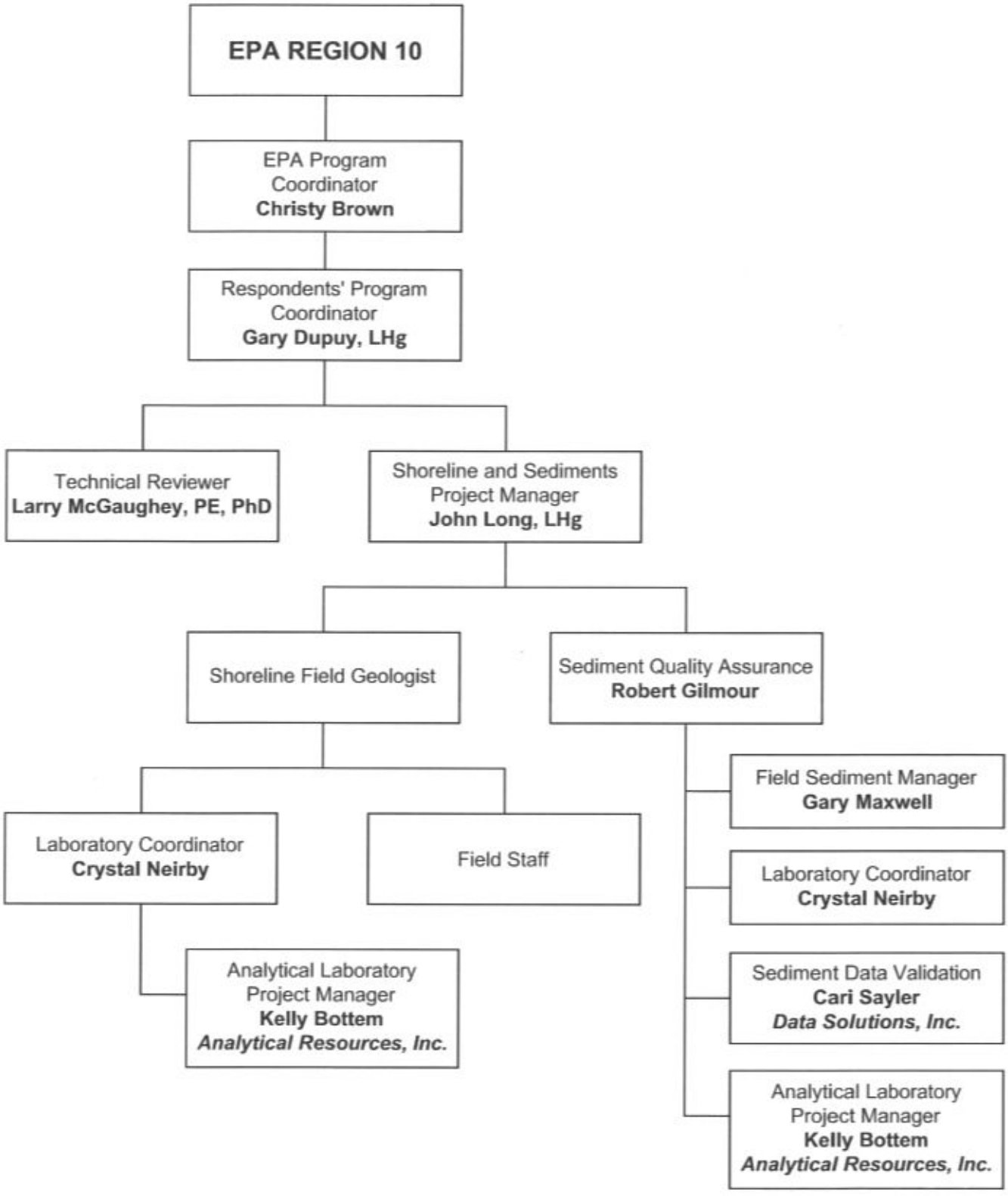
Notes

1. Reporting limits obtained from Analytical Resources, Inc., laboratory.
2. In order to achieve reporting levels below the applicable DQO's in Table 1, the laboratory will report detections between the detection limit and the reporting limit. In the event that carbon normalized non-detected values are greater than the DQO's. The laboratory will evaluate options for reporting lower detection limits such as increasing volume during extraction or analyzing using select ion monitoring. Usability of non-detected results that are greater than the DQOs will be evaluated on a case-by-case basis.
3. Puget Sound Dredged Disposal Analysis protocol for low detection limits.

Abbreviations

µg/kg = micrograms per kilogram
ARI = Analytical Resources, Inc.
CVAA = cold-vapor atomic absorption
DQO = Data Quality Objectives
EPA = U.S. Environmental Protection Agency
HPAH = high-molecular-weight polycyclic aromatic hydrocarbons
ICP-MS = inductively coupled plasma/mass spectrometer
ICP-OES = inductively coupled plasma/optical emission spectrophotometer
LPAH = low-molecular-weight polycyclic aromatic hydrocarbons
mg/kg = milligrams per kilogram
PCBs = polychlorinated biphenyls
PSDDA = Puget Sound Dredged Disposal Analysis
PSEP = Puget Sound Estuary Program

FIGURES



SEDIMENT AND SHORELINE INVESTIGATION
ORGANIZATION CHART
Former Rhone-Poulenc Site
Tukwila, Washington

By: APS	Date: 06/13/11	Project No. 0087690050
AMEC Geomatrix		Figure 1

Plot Date: 06/13/11 - 12:41pm, Plotted by: adam.stenberg
Drawing Path: S:\8769_2006\086_FormerFeatures\, Drawing Name: FRP_FormerSumpSampLoc_060911.dwg

APPENDIX B

Soil and Groundwater Standard Operating Procedures

Analytical Resources, Incorporated
Analytical Chemists and Consultants

Standard Operating Procedure

Metals Analysis – ICP-MS

SOP 538S
Version 007

Revision Date: 4/8/08
Effective Date: 4/8/08

Prepared By:

Jay Kuhn, Jennifer Bouldron

Approvals:

Laboratory / Section Manager

Quality Assurance

4611 South 134th Place, Suite 100 • Tukwila WA 98168 • **206-695-6200** • **206-695-6201** fax

Annual Review

SOP Number: 538S

Title: Metals Analysis – ICP-MS

The ARI employee named below certifies that this SOP is accurate, complete and requires no revisions

Name	Reviewer's Signature	Date

1. Scope and Application

1.1. This Standard Operating Procedure describes the daily operation, tuning, optimization, and analysis procedures for the analysis of samples on an ICP-MS according to EPA Method 200.8 and SW-846 Method 6020. See Appendix 10 for a list of isotopes.

1.2. Most samples will require some form of sample preparation, preservation, filtration and/or digestion, prior to analysis. This procedure is applicable to aqueous samples and acid digestates of solid samples.

1.3. Routine operation and maintenance procedures for the ELAN® 6000 ICP-MS may be found in the ELAN® 6000 Hardware Manual provided by the instrument manufacturer.

1.4. Detailed instructions on the use of the ELAN® 6000 ICP-MS operating software may be found in the ELAN 6000 Software Manual.

2. Summary of the Procedure

2.1. This method describes the multi-element determination of trace elements by Inductively Coupled Plasma – Mass Spectrometry (ICP-MS). Sample material in solution is introduced by nebulization into a radio frequency plasma where energy transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially pumped vacuum interface and separated on the basis of their mass-to-charge ratio. The separated ions are detected and the ion information processed by a data handling system.

2.2. Interferences related to the technique must be recognized and corrected. Such corrections may include compensation for isobaric elemental interferences and interferences from poyatomic ions derived from plasma gas, reagents, or sample matrix.

2.3. Instrumental drift, as well as suppressions or enhancements of instrument response, must be corrected by the use of internal standards.

3. Definitions ERA or NIST

3.1. ICP-MS (Inductively Coupled Plasma - Mass Spectrometer): Refers to an ICP MS spectrometer or to analytical method(s) that specify the use of an ICP-MD to identify and quantify trace elements in environmental samples.

3.2. IDL (Instrument Detection Limit): The concentration equivalent to the analyte signal which is equal to three times the standard deviation of a series of 10 replicate measurements of the calibration blank signal at the selected analytical mass(es).

3.3. RL (Reporting Limit): The RL is the lowest value at which a given analyte is reported. The RL is based on the IDL, MDL, method efficiency, and analysts judgment. The RL will, at minimum, equal

the statistical MDL.

3.4. MDL (Method Detection Limit): As defined in 40 CFR Appendix E Part 136.

3.5. CRDL (Contract Required Detection Limit): Contract specified minimum level of detection.

3.6. ICV (Initial Calibration Verification): A mid-range second source standard, run immediately after calibration to verify the accuracy of the calibration.

3.7. CCV (Continuing Calibration Verification): A mid-range calibration standard, run after every group of 10 samples and at the end of an analytical sequence to verify calibration accuracy during the analytical run.

3.8. ICB (Initial Calibration Blank): A calibration blank run, immediately after the ICV to verify the baseline and to check for carry-over.

3.9. CCB :Continuing Calibration Blank): A calibration blank, run immediately after every CCV to verify the baseline and to check for carry-over.

3.10. QCS (Quality Control Sample): A QC solution supplied by a source independent from the source of the calibration standards. It is used to verify the accuracy of the newly prepared calibration standards.

3.11. IS (Internal Standard): Pure analyte(s) (which is not a sample component) added to a sample, extract, or standard solution in known amounts and used to measure the relative responses of other method analytes that are components of the same sample of solution.

3.12. LR (Linear Range): The linear range of an instrument is the upper limit of accurate quantitation with practical rinse-down times. It varies for each isotope and with instrumental conditions.

3.13. CRI (Low Check Standard): This low level standard is at 1RL.

3.14. SD: Standard Deviation

3.15. RSD (Relative Percent Standard Deviation): The SD divided by the mean, multiplied by 100.

3.16. RPD (Relative Percent Difference): The absolute difference between two numbers, divided by the average of the two numbers, multiplied by 100.

3.17. %R (Spike Percent Recovery): The difference between the matrix spike concentration and the background sample concentration divided by the concentration of the spike added multiplied by 100.

3.18. Analytical Batch: An analytical batch shall consist of no more than 20 samples.

3.19. MB (Method Blank.): An aliquot of analyte-free matrix taken through the sample preparation procedure with each analytical batch.

3.20. LCS/MBSPK (Laboratory Control Sample, Reference Sample or Method Blank Spike): A reference solution of known concentration processed along with the analytical batch to test the

digestion procedure for accuracy. Both soil references and aqueous references (reference solutions or method blank spikes) are LCSs.

3.21. SRM (Standard Reference Material): A reference sample of known concentration processed along with the samples to test the digestion procedure for accuracy. For a soil sample the reference used is typically an ERA Soil SRM.

3.22. MS (Matrix Spike): A sample prepared by adding a known amount of analyte to a specified amount of sample matrix. Matrix spikes are used to determine the effect of the sample matrix on the method's recovery efficiency.

3.23. MSD (Matrix Spike Duplicate): A second replicate matrix spike sample prepared and analyzed as above (Sec. 3.21) to measure precision with respect to a given matrix.

3.24. MD (Matrix Duplicate): A second replicate matrix sample prepared and analyzed with a sample batch to measure precision with respect to a given matrix.

3.25. ICSA, ICSAB (Interference Check Solutions): The ICSA solution contains interfering elements, at levels often found in samples, to test the efficacy of instrument correction equations. The ICSAB solution contains the same elements as the ICSA at the same concentrations, plus analytes at moderate levels to test the accuracy of analyte measurement in the presence of interferents.

3.26. Carry-over: The effect of a high level sample on a lower level sample which follows. Residual analyte from the high level sample may remain in the uptake lines, in the nebulizer, in the spray chamber or in the torch. The lower level sample which follows may or may not show successively decreasing exposures as evidence of carry-over. A true test for carry-over is the analysis of a blank, which has either elevated analyte or successively decreasing exposures. Carry-over may also be referred to as memory effect. If carry-over is suspected, the lower level sample should be rerun following a blank, another low level sample, or an extended rinse time.

3.27. Tuning Solution: A solution which is used to determine acceptable instrument performance prior to calibration and sample analyses.

3.28. Stock Standard Solution: A concentrated solution containing one or more method analytes prepared in the laboratory using high purity solutions purchased from a reputable commercial source (Inorganic Ventures).

3.29. Analysis Protocols:

3.29.1. Routine or Package: Follows SW-846 6020 and EPA 200.8. If the analysis comment is Package (PKG), a data package will be generated from the Routine analytical run and Routine QC samples. Other modifications that are necessary for certain quality assurance plans and/or agencies will be detailed in individual sections.

3.29.2. CLP-Q: Follows Routine protocol with CLP-type and DOD QC standards analyzed at CLP QC frequency, in order to generate a CLP-type data package.

4. Interferences

4.1. Isobaric interference occurs when an isotope of one element is at the same nominal mass as an isotope of another element (e.g., ^{98}Mo and ^{98}Ru). Corrections for isobaric interference may be made by measuring the intensity due to the interfering element at another isotope and using its natural abundance ratios to correct for its presence at the analytical mass of interest. Most commonly used corrections for isobaric interference are already present as default interference equations in the ELAN® NT software. Care should be taken that the isotope measured for correction purposes does not suffer from overlap with other isotopes that may be present in the sample.

4.2. Molecular interferences are caused by molecular species formed in the plasma with argon plasma or matrix ions (examples of common molecular interference include ArCl, ClO, nitrogen dimer, oxygen dimer, oxide species, double charged species, etc.). Predictions about the type of molecular interference may be made using knowledge about the sample matrix. Molecular interference can often be corrected for in the same manner as isobaric interference, i.e., measuring the intensity present at another isotope and using isotope ratios to calculate the amount of the interfering species. For example, corrections for interference of $^{40}Ar^{35}Cl$ on As at mass 75 may be made by measuring the intensity of ArCl present at mass 77 ($^{40}Ar^{37}Cl$) and converting to the apparent intensity of ArCl at mass 75 by using the isotopic ratio of ^{37}Cl to ^{35}Cl. A list of the correction equations used is given in Appendix 9.

4.3. Physical interferences are associated with solution viscosity and surface tension differences between standard solutions and samples. These interferences may occur in transfer of solution to nebulizer, at the point of aerosol formation and transport to the plasma, or during the excitation and ionization process within the plasma. Internal standardization is used to compensate for many physical interference effects. Five internal standards are chosen to closely match the analytical behavior of the elements being determined.

4.4. Memory Interferences: Result when isotopes of elements in a previous sample contribute to the signals measured in a new sample (Carry-over as defined in Section. 3.26). If a memory interference is suspected, the sample should be reanalyzed after a long rinse period.

5. Safety

5.1. The use of laboratory equipment and chemicals exposes the analyst to several potential hazards.

Good laboratory technique and safety practices shall be followed at all times.

5.2. Safety glasses shall be worn at all times when handling samples, reagents, or when in the vicinity of others handling these items.

5.3. Liquid argon represents a potential cryogenic hazard and safe handling procedures shall be used at all times when handling liquid argon tanks.

5.4. The ELAN® 6000 is fully interlocked to protect the user from dangers such as high voltages, radio frequency generators, and intense ultra-violet light. At no time should the operator attempt to disable these interlocks or operate the ELAN® 6000 if any safety interlock is known to be disabled or malfunctioning.

5.5. Spilled samples, reagents, and water shall be cleaned up from instrument and autosampler surfaces immediately.

5.6. All additional company safety practices shall be followed at all times.

6. **Equipment and Supplies**

6.1. Perkin-Elmer ELAN® 6000 ICP-MS system: includes the ELAN® 6000 instrument, a peristaltic pump, a computer system, ELAN® NT software, a printer, and a Perkin-Elmer autosampler with a dust cover.

6.2. Supplies

6.2.1. Peristaltic pump tubing:

6.2.1.1. Black/Black Tygon- 0.76 mm id used for sample introduction

6.2.1.2. Red/Red Tygon – 0.80 mm id used for rinse station

6.2.1.3. Black/White Tygon- 3.16 mm id used for the spray chamber drain

6.2.2. Calibrated mechanical pipettes with metal-free plastic pipette tips

6.2.3. 15 mL and 50 mL polypropylene metal-free auto-sampler tubes with caps

6.2.4. 100, 200, 500 and 1000 mL polyethylene volumetric flasks

7. **Reagents and Standards**

7.1. Reagents

7.1.1. All reagents may contain impurities that may affect the integrity of the analytical results. Due to the high sensitivity of ICP-MS, high purity reagents, water, and acids must be used whenever possible. All acids used for this method must be of high purity grade. Nitric acid is preferred for ICP-MS in order to minimize polyatomic interference.

7.1.2. Nitric acid (HNO_3), concentrated. Trace Grade HNO_3 that has Lot QC documentation to verify that it is acceptable for trace metals use.

7.1.3. Hydrochloric acid (HCL), concentrated, Trace Grade HCL that has Lot QC documentation to verify that it is acceptable for trace metals use.

7.1.4. Deionized water (DI). Type I reagent water (18.3 megaohm).

7.1.5. 1% (vol/vol) nitric acid. Add 10 mL of trace grade nitric acid to a 1 liter volumetric flask containing 900 mL of DI water. Mix well and bring to volume.

7.2. Standards: All standards must be labeled with the analyst's initials, preparation date, expiration date, and standard identification number (from the Metals Working Standard Preparation Log). The preparation of all standards and analytic solutions must be documented in the Working Standards Log. Standards are stored in the metals lab either on the standard shelves or near the point of use. All standards are marked with an expiration date derived from the expiration date of the stock from which it is made, or from method requirements.

7.2.1. Single element stock standards of the elements at the highest purity available form Inorganic Ventures. All single element standards and Intermediate Standards are checked by ICP and/or ICP-MS prior to use.

7.2.2. Tuning Solution:

7.2.2.1. Tuning Solution Intermediate (1 mg/L Be, Mg, Co, In, Ba, Ce, and Pb in 1% HNO_3): Prepare by pipetting 0.1 mL of each 1000 mg/L single element stock standard into a 100 mL volumetric flask containing 90 mL DI water and 1.0 mL concentrated HNO_3. Dilute to 100 mL with DI water and mix well (see Appendix 1 Section 2).

7.2.2.2. Working standard (10 µg/L Be, Mg, Co, In, Ba, Ce, and Pb in 1% HNO_3): Prepare by pipetting 10.0 mL of Tuning Solution Intermediate into a 1000 mL volumetric flask containing 900 mL of DI water, and 10 mL of concentrated HNO_3. Dilute to 1000 mL with DI water and mix well.

7.3. Internal Standard Solution:

7.3.1. Solution Preparation (30 mg/L ^{6}Li, 10 mg/L Ge, 2 mg/L Sc; and 1 mg/L Y, In, Tb and Bi): Prepare by pipetting 15 mL ^{6}Li, 5.0 mL Ge, 1.0 mL Sc, and 0.5 mL each Y, In, Tb, and Bi of 1000 mg/L individual stock standards into a 1000 mL volumetric flask containing 800 mL DI water and 10 mL concentrated HNO3. Dilute to 1000 mL with DI water and mix well (see Appendix 1 Section 1).

7.3.2. Add Internal Standard Solution (at a ratio of 1ml Internal Standard Solution to 100 mL final solution volume) to all standards and samples before analysis.

7.4. Calibration Standard Stocks (see Appendix 2):

7.4.1. Calibration Stock 1 - containing 100 mg/L Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Ni, Pb, Se,

Tl, Th, U, V, and Zn.

7.4.2. Calibration Stock 2 - containing 100 mg/L Mo and Sb.

7.4.3. Individual Stock Standards each containing 10,000 mg/L of Al, Ca, Fe, K, Mg, and Na

7.5. Calibration Intermediate Standard (See Appendix 3) is prepared, as needed, by adding 10.0 mL of each of the Stock Standards 1 and 2, and the individual elements of Al, Ca, Fe, K, Mg, and Na into a 100 mL volumetric flask containing 10 mL DI water and 1.0 mL of concentrated HNO_3. Bring to volume with DI water and mix well.

7.6. Calibration working standards: Prepare fresh every two weeks or as needed at the specified concentrations in Appendix 4. All solutions, including calibration blanks, calibration standards, samples, and quality control standards or samples should be spiked with 1 mL of the Internal Standard solution for each 100 mL of solution prior to analysis.

7.7. QCS: The QCS, typically an ERA or NIST solution, is prepared as recommended by the supplier. It is diluted appropriately and analyzed as a sample when required (see section 15.5).

7.8. Initial Calibration Verification Standard: This is a second source calibration verification standard prepared from 2 second source stock solutions purchased from Inorganic Ventures Inc. (see Appendix 5). Prepare by pipetting 20.0 mL of AR-ICVMS-1 stock and 20.0 mL of AR-ICVMS-2 stock into a 1000 mL volumetric flask containing 900 mL of DI water, and 10 mL of concentrated HNO_3. Dilute to 1000 mL with DI water and mix well. After diluting to volume, add 10mls of Internal Standard Solution and mix well.

7.9. Low Check Solution (CRI): A solution prepared at the RL for each element to check the accuracy at low levels.

7.9.1. Low Check Intermediate (see Appendix 7): Prepare by pipetting the specified amount of single element stock standard into a 100 mL volumetric flask containing 80 mL DI water and 1.0 mL concentrated HNO_3. Dilute to 100 mL with DI water and mix well.

7.9.2. Low Check Solution (levels at 1RL): Prepare by pipetting 0.05 mL of Low Check Intermediate into a 100 mL volumetric flask containing 90 mL of DI water, and 1.0 mL of concentrated HNO_3. Dilute to 100 mL with DI water and mix well. After diluting to volume, add 1ml of Internal Standard Solution and mix well.

7.10. Continuing Calibration Verification: Standard 3 (50 µg/L non-minerals and 5000 µg/L minerals), the mid-range calibration standard, is used.

7.11. Calibration Blank: A solution containing 1% (v/v) concentrated HNO_3 in DI water. Fill a 1-L volumetric flask with approximately 900 mL of DI water. Pipette 10 mL of concentrated HNO_3 into the flask, dilute to 1000 mL with DI water and mix well. After diluting to volume, add 10mls of

Internal Standard Solution and mix well.

7.12. Dual Detector Calibration Solution: contains 250 µg/L each of Ag, Al, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Th, Tl, U, V, Zn, ^{6}Li, Sc, Ge, Y, In, Tb, and Bi (see Appendix 6).

7.12.1. Dual Detector Calibration Intermediate Standard: Prepare by adding 0.5 mL of each of the above 1000 mg/L elements into a 200 mL volumetric flask containing 150 mL DI water and 2 mL concentrated HNO3 . Dilute to 200 mL with DI water and mix well. Note: There is no need to add Se or additional internal standards to this solution.

7.12.2. Dual Detector Calibration Working Standard: Add 100 mL of the dual detector calibration intermediate standard into a 1000 mL volumetric flask containing 800 mL DI water and 10 mL conc. HNO_3. Dilute to 1000 mL with DI water and mix well.

7.13. Interference Check Solutions (ICSA, ICSAB): These solutions are made from Inorganic Ventures multiple element stock standards (see Appendix 8). Prepare weekly.

7.13.1. ICSA: Prepare by pipetting 20.0 mL of Inorganic Ventures AR-6020ICS-OA10 stock into a 100mL volumetric flask containing 70 mL of DI water, and 1.0 mL of concentrated HNO_3. Dilute to 100 mL with DI water, add 1.0 mL Internal Standard solution and mix well.

7.13.2. ICSAB: Prepare by pipetting 20.0 mL of Inorganic Ventures AR-6020ICS-OA10 stock and 1.0 mL of ICP-MS ICSAB stock into a 100 mL volumetric flask containing 70 mL of DI water, and 1.0 mL of concentrated HNO_3. Dilute to 100 mL with DI water, add 1.0 mL Internal Standard solution and mix well.

7.13.2.1. ICP-MS ICSAB STOCK: Prepare by pipetting 0.10 mL each of 1000mg/L Stock standards Ag, As, Cd, Co, Cr, Cu, Mn, Ni, Zn into a 50 mL volumetric flask containing 20 mL of DI water and o.5 mL of concentrated HNO3. Mix well and transfer to a 60 mL clean Nalgene container.

8. Sample Collection, Preservation, Shipment and Storage

8.1. All samples should be received in appropriate collection containers and have been properly preserved by the clients.

8.2. Samples are checked for proper preservation and stored refrigerated for a maximum of 180 days prior to sample preparation.

8.3. Some samples are shared with the organic extractions and or conventionals laboratories. Sample receiving places these samples in a share bin in Refrigerator 5. SOP 1019S includes procedures for handling shared samples.

9. Quality Control

9.1. Documentation

9.1.1. Instrument logbooks

9.1.1.1. Daily analysis: The ICP-MS Sample logbook shall be used as a run sequence log, for sample specific notes, for QC limit notes, and for any notes pertaining to the run.

9.1.1.2. Maintenance logbook: Shall be used for notes of periodic checks of instrument performance and of all maintenance procedures including physical changes (tubing, cones etc.) and operational maintenance routines (i.e. Dual Detector Calibrations)

9.1.2. ICP-MS Files

9.1.2.1. Daily Tuning , Performance, and AutoLens® results are filed daily.

9.1.2.2. Dual detector summary results are filed with that days Tuning, Performance and Auotlens.

9.1.2.3. Standard Certificates: Contains a Certificate of Analysis for all Inorganic Venture Standards as well as standards obtained from other sources.

9.2. Calibration Standards: All standards are prepared every two weeks (or as needed) by dilution from known intermediates and verified by the analysis of certified second-source QC standards (Section 7.4-7.6).

9.2.1. Calibration verification must be performed immediately after calibration with an Initial Calibration Verification Standard (ICV) and after every 10 samples and at the end of the analytical run with a Continuing Calibration Verification Standard (CCV). The ICV is made from a source other than that used for the preparation of the standard curve. The CCV is the mid-range Calibration Standard.

9.2.2. Calibration Verification Blanks (ICB/CCB) are analyzed to confirm the absence of blank contamination, baseline drift and/or carryover. Immediately after the ICV and every CCV a Calibration Verification Blank must be run.

9.2.3. Independent QC solutions: This standard is used to check the calibration stock standards stability, concentrations, and preparation. They are analyzed on the day new calibration standards are prepared.

9.3. Low Check (CRI) Standard is analyzed following calibration verification for each element. The standard is used to verify the analytical performance at the low end of the calibration or reporting limit.

9.4. Interference Check Solutions (ICSA/B) The interference check solutions (See Appendix 8) are analyzed to check the accuracy of correction equations. These standards are analyzed after the

Low Check Standard.

9.5. Serial Dilution: A five-fold should be performed on any new or unusual sample matrix. This dilution test will help identify a matrix interference if one is present. The dilution is performed on a sample, typically the sample used for matrix QC, from each group of samples of a similar prepcode for each sample digestion batch. Some projects, including DOD, require a serial dilution be performed on at least one sample in their batch.

9.6. Post Digestion Spike: A post digestion spike should be performed on a new or unusual sample matrix, along with the serial dilution. This sample is intended to help identify matrix interference problems. Some projects, including DOD, require that a post digestion spike be performed on at least one sample in their batch. For CLP type samples this spike is required only for elements that are outside control limits in the Matrix Spike sample. For DOD samples all requested elements must be spiked.

9.7. Matrix QC samples: With each preparation batch various matrix QC samples must be analyzed. At a minimum a matrix spike, matrix duplicate, method blank, and a laboratory control sample should be prepared and analyzed. For some projects matrix spike duplicates, laboratory control sample duplicates, and/or certified reference materials may also be required. The analyst must check all paper work to make sure all necessary QC samples have been prepared and analyzed.

9.8. All logbooks are reviewed monthly for completeness and accuracy by laboratory personnel.

9.9. The QA section will periodically review the standard preparation process, including standard bottles, logbooks and standard certificates and traceability to standardized sources.

9.10. Initial Demonstration of Laboratory Performance - The following items must be completed before the analysis of any samples is performed by using this method

9.10.1. Instrument Detection Limits (IDLs) shall be determined for all analytes at quarterly.

9.10.2. Calibrate the instrument, and then run the usual QC sample sequence.

9.10.3. Run the blank solution as a series of 10 sequential samples with rinsing in between each sample.

9.10.4. Calculate the standard deviation of the 10 blank samples for each isotope.

9.10.5. IDLs shall be re-determined quarterly or following any significant change to the instrument (new detector or different sample introduction system used).

9.11. Method detection limits (MDLs) shall be established for all analytes by the method outlined in 40 CFR Part 136 at least annually.

9.11.1. Fortify a reagent blank with a concentration of each analyte that is two to five times the estimated detection limit (the IDL can be used to estimate this). This solution is called the

MDL solution.

9.11.2. Take eight replicate aliquots of this solution and process through the entire method, including any sample preparation steps. Run these as samples with rinsing between each sample. Calculate the standard deviation of the samples for each isotope. Multiply the standard deviation by 2.998 (student's t value for 99% confidence level and n=8) to obtain the MDL.

9.11.2.1. The MDLs must be lower than in-house RLs. If not, the RLs will need to be changed or the MDL analysis redone.

9.11.2.2. MDLs shall be re-determined annually or following any change to the sample preparation procedure

10. **Calibration and Standardization**

10.1. The instrument must be calibrated using a blank and 4 calibration standards before analysis. The high concentration standard will contain of 100 µg/L of all analytes except for the minerals (Al, Ca, Fe, K, Mg, and Na) which will be at a concentration of 10,000 µg/L.

10.1.1. The concentrations of the standards have been entered into the calibration page of the analytical method in the ELAN® software according to the values of the standards prepared in Section 7.6.

10.1.1.1. A "Linear Through Zero" curve type should be selected for all analytes.

10.1.1.2. The calibration blank should be run as a blank, before the analysis of any calibration standards.

10.1.1.3. The first standard run should be the lowest level standard, followed by standards of increasing concentration in order to minimize cross-contamination and carry-over.

10.2. Load the calibration blank and the calibration standards into the autosampler positions specified on the Sampling page of the analytical method.

10.2.1. Start calibration in the Samples window by highlighting the 1st row of the batch labeled "Rinse Sample" with calibration action "Run Blank, Standards, and Samples". Click on Analyze Batch. Click on Yes to clear the previous calibration.

10.2.2. After calibration has run, save the calibration file. Review the data for acceptable RSDs and internal standard recoveries (see Section 15). View the curve-fit on the Calibration View page for poor curve fit (apparent standard levels >5% from true value), then print a calibration summary. To print a calibration summary, click on the Report page, and under Report View, Report Options Template select **arical.rop**. Then click on the Dataset Icon, highlight the latest calibration standard row and click on Reprocess. Review the r-values for any that are

<0.9990. If poor curve fit and/or poor r-value are found, rerun a standard or recalibrate (also see Section 1.1). To reset the usual report options template, click on the Report page, and under Report View, Report Options Template select **ariquant.rop**.

10.3. Analyze Required QC Samples

10.3.1. Analyze the ICV and ICB standards. Confirm the standard recovery is within 10% of the known value, and the ICB value is less than the RL (unless analyzing DOD samples, when the CB value must be less than 2X MDL). If these conditions are not satisfied the analysis must stop, the problem corrected, the instrument recalibrated and the Initial Standards rerun.

10.3.2. Analyze Low Check Standard (CRI). Confirm the standard is within 50% of the known value (20% for DOD), else the problem must be corrected and the CRI run within limits before samples may be analyzed.

10.3.3. Analyze ICSA/B Standards. Confirm the standards return values of less than the RL for all non-spiked elements (unless contamination can be documented), and within 20% of the known value for spiked analytes. (For DOD the absolute value for all non-spiked analytes shall be <2XMDL, unless a verified trace impurity from one of the spiked analytes exists.)

10.3.4. Analyze the independent QC solution(s) if fresh calibration standards were prepared on this day. This standard serves as an additional check of the calibration solutions. The concentrations should be within the certified limit range provided by the supplier. If the results are outside this range, the analyst should compare the percentages to the nearest CV and rerun the QC solution, recalibrate, or prepare new calibration standard(s).

11. **Procedure**

11.1. Initial Demonstration of Laboratory Performance: The following items must be completed before the analysis of any samples is performed using this method.

11.1.1. Instrument Detection Limits (IDLs) shall be determined for all analytes quarterly. Detection limits established shall be less than or equal to the MDL.

11.1.1.1. Calibrate the Instrument, and then run the usual QC sample sequence.

11.1.1.2. Run the blank solution as a series of 10 sequential samples with rinsing in between each sample.

11.1.1.3. Calculate the standard deviation of the 10 blanks for each isotope.

11.1.1.4. IDLs shall be determined whenever the following occurs:

11.1.1.4.1. Quarterly

11.1.1.4.2. Any significant change to the instrument (ie. new detector or a different sample introduction system used)

11.1.2. Method detection limits (MDLs) shall be established for all analytes by the method outlined in 40 CFR Part 136, at least annually.

11.1.2.1. Fortify a reagent blank with a concentration of each analyte that is two to five times the estimated detection limit (the IDL can be used to estimate this). This solution is called the MDL solution.

11.1.2.2. Take eight replicate aliquots of this solution and process through the entire method including sample preparation steps. Run these as samples with rinsing between each sample. The resulting values are entered into the spread sheet for MDL calculations. Following the MDL determination an MDL verification check sample may be run (spiked at approximately 2XMDL). The MDL check must produce a response at least 3X instrument noise level.

11.1.2.3. The MDLs must be lower than the in-house RLs. If not, the RLs will need to be changed or the MDL analysis redone.

11.1.2.4. MDLs shall be redetermined whenever the following occurs:

11.1.2.4.1. Annually

11.1.2.4.2. Any change to the sample preparation procedure.

11.1.3. Linear Range Verification: Linear range limits are determined during the methods development period. Many of them are set below instrument capability, limited by other concerns such as carry-over and rinse-out times. Linear Limits used are verified on an on-going basis and at least every six months. Linear Range standards (LR200, LR300) are analyzed to verify each element is within 10% of expected value and results may be reported to that level.

11.2. Daily Procedure

11.2.1. Preparing the uptake system and interface

11.2.1.1. Open the instrument cover and slide the vacuum chamber away from the torch using the lever. Check the condition of the cones for deposits and wipe the sampler cone with a Kimwipe® wetted with a small amount of DI water. When deposits are severe, change or clean the cones.

11.2.1.2. Attach the three pump tubing to the peristaltic pump. Black-black tubing for the sample line, red-red for the rinse station line and black-white tubing for the spray chamber drain line. The rinse tubing can be left unclamped until the pre-analysis routines are complete. Make sure the tubing is not flattened, or else change to new sample and drain tubing for optimum performance. Attach a length of PFA tubing and insert it in DI water.

11.2.1.3. Initiate the plasma and allow a warm-up of at least 45 minutes. Ensure that the spray chamber is pumping out smoothly.

11.2.2. Open the **aritune.wrk** workspace to perform the tuning.

11.2.2.1. Manually aspirate (without the autosampler) the 10 µg/L Tuning solution (see Section 7.2.2.2). This solution will be continuously aspirated for the tuning, the AutoLens® calibration and the daily performance check.

11.2.2.2. Click on the Tune Mass Spec button in the Tuning window.

11.2.2.3. After the tuning solution has run, print a tuning report for each of the tunings performed by clicking on the Printer icon. Print each tuning before starting the next tuning.

11.2.2.4. Check that the mass calibration for each of the measured masses is ± 0.05 AMU of the true mass. Methods 6020 and 200.8 require ± 0.1 AMU.

11.2.2.5. Check that the resolution for all elements is 0.7 ± 0.03 AMU (measured at 10 % peak height). EPA Method 6020 requires 0.9 amu at 10% peak height; Method 200.8 requires 1 amu at 5% peak height.

11.2.2.6. . If both the resolution and mass calibration are acceptable, save the file to disk.

11.2.2.7. If any of the tuning parameters are not meeting method specifications and the instrument requires adjustment, see Section 3 of the ELAN® 6000 Software Manual. It is typical to run several tunings to get all 5 elements within specifications.

11.2.3. Open the **ariAutoLenscal.wrk** workspace to perform the AutoLens® calibration.

11.2.3.1. Continue to aspirate the 10 µg/L tuning solution.

11.2.3.2. Clear the old calibration by clicking on Clear Calibration.

11.2.3.3. Click on Get Analyte List

11.2.3.4. Click on Calibrate (the procedure takes about 6 minutes).

11.2.3.5. Print an Interactive Graph (add all elements) and save the AutoLens® Calibration.

11.2.3.6. Review the AutoLens® calibration graphs; irregularly shaped peaks or voltages above 10 may necessitate lens cleaning (see section 1.2.4).

11.2.4. Open the **aridailyperf.wrk** workspace to perform the daily performance check.

11.2.4.1. . Continue to aspirate the 10 µg/L tuning solution.

11.2.4.2. . Click on the Analyze Sample button in Sample window to start analysis.

11.2.4.3. . Check that the RSDs for five replicates for Ba, Ce, In, Mg, and Pb are all ≤5% as required by EPA Methods 6020 and 200.8 (typically all are <3%).

11.2.4.4. Monitor the daily performance measures (as recommended by Perkin Elmer) of Mg, In and Pb sensitivity, background, % double charged and % oxide levels:

11.2.4.4.1. Mg at > 40,000 cps, In at 10 ppb > 300,000 cps, and Pb at 10 ppb > 150,000 cps

11.2.4.4.2. Background at mass 220 < 30 cps

11.2.4.4.3. Ba^{+2} <0.03 (% double charged < 3%)

11.2.4.4.4. CeO <0.03 (% oxides < 3%)

11.2.4.4.5. Oxides and double charged levels can be reduced by slightly decreasing the nebulizer flow rate or increased by slightly increasing the nebulizer flow rate. To adjust the flow, click on the **Optimize** Icon, **Manual Adjust**, and then adjust the nebulizer gas flow arrows: try using 0.01-0.02 increments to adjust CeO from 0.26 to 0.28.

11.2.4.4.6. Low sensitivity for Mg (representative of low masses) alone may indicate the need for lens cleaning (see Section 1.2.4)

11.3. Sample Analysis

11.3.1. Open the **2008ARI.wrk** workspace and load the applicable method for the analytes required (see the method list at the instrument). In the Method window, click on the Report page. Change the report filename to reflect the run date **msyymmdd.rep**, where ms is the analysis method, yy are the last 2 digits of the year, mm is the month and dd is the day of the month). Save the method file.

11.3.2. New sample types can be screened by ICP or the semi-quantitative ICP-MS procedure, Total Quant. To screen a sample using Total Quant, first open the **ARITQ.wrk** workspace. Calibrate using the 1% HNO3 diluent as the calibration blank and standard 3 (50 µg/L non-minerals and 5000 µg/L minerals) as the single calibration standard. Run an ICV and an ICB before running samples at an appropriate dilution; new sample types are diluted at least 1/10, more dilute (1/50 or 1/100) if warranted.

11.3.3. Samples Analysis Setup

11.3.3.1. Edit the Samples window to enter new samples in the autosampler sequence.

11.3.3.2. Click on Batch: The starting sample sequence is ICV, ICB, CCV1, CCB1, Low Check, ICSA, ICSAB, CCV2, CCB2 and then 10 client samples. 10 samples can be run between CCV/CCB pairs (Low Check, ICSA and ICSAB count as samples). Run the QCS (if necessary) early in the run. Linear range checks, LR200 and LR300, can be run anytime after the Low Check, ICSA, ICSAB group. Enter the autosampler sample position in the A/S Loc column. Enter the dilution factor in the Batch ID column. REN preps are diluted ½ to start and RHN preps are run undiluted. SWN preps are diluted 1/20. Enter the sample identification in the Sample ID column using the standard format: client job (space)

sample letter (space) prepcode (e.g. A001 B REN). The calibration action for all samples is "Analyze Sample".

11.3.3.3. Enter peristaltic pump control speeds for all samples: -24 for normal speed. Enter the number of seconds required for sample flush, read delay and wash. Especially watch these fields when adding sample rows, they will remain empty until edited.

11.3.3.4. Check the autosampler positions.

11.3.3.5. Rinse 15ml metals free tubes in groups of six. Rinse with 10% Nitric Acid followed by Di. Add the appropriate Internal Standard solution to each tube (For 6 mL total volume of sample add 0.6mL of I.S). Prepare the sample dilutions according to sample prep method and knowledge of sample analyte levels. Mix the samples using the small vortexer.

11.3.3.6. Load the samples into the autosampler positions specified in the sample table. Save the sample file.

11.3.3.7. Select the samples to be analyzed by highlighting the rows.

11.3.3.8. Click on Analyze Batch. A Pop-up window will ask if you wish to clear the current calibration – Click on NO unless you wish to recalibrate Click on Yes will clear the current calibration).

11.3.4. During the run, the instrument condition and sample results are monitored so appropriate actions can be performed as needed. The CVs, CBs, QC solutions, Linear Range standards, MBs, LCSs, duplicates, matrix spikes, and internal standard recoveries should be checked during the run or soon thereafter. See Sction 15 for acceptance criteria.

11.3.5. Analytical Run Order

11.3.5.1. Routine: After acceptable initial QC has been run, every group of 10 samples must be preceded and followed by a CCV, CCB pair. Typically each group of ten may be started with suspected low level samples, such as matrix blanks and ending with higher level samples such as reference materials, or matrix spikes. In order to facilitate acceptance limit checking samples may be run: matrix duplicate, background sample, matrix spike, reference sample.

11.3.5.2. CLP-Q/DOD The analytical run order is as above plus: a serial-dilution (a five fold dilution must agree within 10% of the undiluted sample). A post-digestion spike is performed on DOD samples with recovery 75-125% of the expected result.

11.3.6. Monitoring the Analysis

11.3.6.1. Periodically during the run check the sample uptake flow, the level of solution in the autosampler QC vials, the autosampler probe position and the rinse bottle level. To pause

the run, click on 'CANCEL' (to stop immediately) or 'STOP' (to stop after current sample)

11.3.6.2. Method QC samples such as CV/CB should be monitored closely during the analytical run to check for calibration stability and baseline drift. If the CV and/or the CB are outside or approaching the QC limits, then corrective action should be taken as soon as possible to minimized sample reruns. Corrective action could include recalibrating the Blank or extra rinsing time followed by another CV and CB.

11.3.6.3. Monitoring High Levels and Carryover: If any sample level is above the linear limit, the sample should be diluted and rerun for the affected element. If high level samples are analyzed, carryover into the following samples may occur. Carryover usually exhibits high SD or RSD in the following samples. If carryover is suspected the affected sample should be reanalyzed. Note: Some projects (including DOD) require any sample analyte that exceeds the concentration of the high calibration standard be diluted. If required, appropriate instructions will be in the job folder and it is the analysts responsibility to follow the instructions and document the necessary dilutions on the raw data.

11.3.7. Recalibrating the Calibration Blank: (It is possible to recalibrate the Blank and re-set the internal standards during a run):

11.3.7.1. Stop the run and open the Manual tab on the sample table.

11.3.7.2. Click on the Analyze Blank option. After the blank has run return to the Batch Sample Mode. Any recalibration of the Blank must be followed by a CV/CB within QC limits before samples may be run.

11.3.8. Shut Down

11.3.8.1. Aspirate 5% HNO_3 for 10 minutes, and then aspirate DI water for 10 minutes. Move the probe to "Standby" to drain the uptake lines and the spray chamber. When the spray chamber drain line is empty, extinguish the plasma. Loosen the peristaltic pump tubing.

11.3.8.2. Auto-shutdown (unattended shutdown) requires adding a 5% HNO_3 sample (use autosampler position 7) and a DI water sample row (use autosampler position 6) to the last autosampler sequence. Set for a 600 sec sample flush at –24 pump speed, 0 sec delay, and 0 sec wash. In the Instrument window, click on Auto Start/Stop and click on Enable under Auto Stop. Set Auto Stop for 0 second delay. Auto-shutdown MUST be set prior to starting the batch analysis! Start the autosampler batch. On the following workday, run the peristaltic pump to completely drain the uptake lines and the spray chamber, then loosen the pump tubing. Allow the pump tubing to relax for at least 1 hour. Alternatively, the tubing can be removed and saved in a bag labeled as "used" and new tubing installed.

12. Data Analysis and Calculations

12.1. Data Entry

12.1.1. To transfer the data file to the Network: On the desktop, double click on the Report Output folder. Highlight the filename and drag it to the Network folder.

12.1.2. To archive raw data files: Every few runs, move the raw sample files (except those from the most recent run) from the C drive the sub-directory Elandata\Dataset\Default to the "annual" Archive. On the desktop, double click on the Default folder. Highlight the files and drag them to the "annual" folder.

13. Method Performance

13.1. An Instrument Detection Limit (IDL) Study is performed once each quarter. IDL summaries are maintained by ARI's QA Section and available for review upon request. IDL values are ≤ the MDL for all analytes.

13.2. MDL studies are performed for all analytes as described in Section 9.11.

13.2.1. The MDLs must be lower than in-house RLs. If not, the RLs will need to be changed or the MDL study replicated.

13.2.2. MDLs shall be re-determined annually or following any change to the sample preparation procedure

13.3. Analytical accuracy is determined using LCS/MBSPK, SRM or MS analyzes. Acceptance limits for spike recovery are specified in the analytical methods and are normally 80 to 120% for LCS and 75 to 125% for matrix spikes. Acceptance limits for SRM analyzes are determined by the SRM supplier or manufacturer.

13.4. Laboratory precision is measured by performing replicate analyzes. Replicates (sample or matrix spike) acceptance limits are ± 20%.

13.5. Accuracy and precision acceptance limits are disseminated to the bench chemists and LIMS administrator for use in monitoring method performance in real time.

14. Pollution Prevention

14.1. All acidified sample waste must first be neutralized prior to sink disposal.

14.2. Dispose of expired standards into the designated barrel in the hazardous waste room.

14.3. Samples that are designated as hazardous waste by the LIMS "Hazardous Report" must be placed in the designated drum in the Hazardous Waste Storage Area when they are disposed. This process is described in SOP 1003S.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Precision Criteria: RSDs should be ≤5% for concentrations ≥10RLs, SD ≤1RL for concentrations <10RLs. If a sample has poor precision (RSD or a SD outside these limits), rerun the sample. If the RSDs or SDs are still outside the limits, then the sample may need to be rerun at dilution (if no instrumental precision problems are suspected).

15.2. Internal Standard Responses

15.2.1. The internal standard intensities for all internal standards used will be monitored and compared to the intensity in the most recently run calibration blank.

15.2.2. The intensities of the internal standards in all samples, QC, and continuing calibration checks should be 60-120% of the original response in the calibration blank. If the responses are not within the limits, rinse for 10-15 minutes, then run a CCB to check the intensities of the internal standards in the blank. If the intensities are now close to the intensities of the internal standards in the original calibration blank, dilute the sample by a factor of 2 to 5, and reanalyze.

15.3. Isotope Selection:

15.3.1. Look for significant differences (>2% for concentrations ≥50RLs, or >1RL for concentrations <50RLs) between isotopes on those elements which have multiple isotopes. If the difference is significant, choose the isotope with the lowest concentration.

15.3.2. If As or Se is required, evaluate the difference between ^{78}Se and ^{82}Se. If ^{82}Se is significantly higher (≥ 2RL) than ^{78}Se, bromide interference is indicated, and ^{78}Se must be used. ^{78}Se is noisier and more susceptible to baseline drift than ^{82}Se; watch the precision and the baseline checks. When ^{82}Se is used, report the As#1 result (1st As value listed) which has an ^{82}Se correction equation. When ^{78}Se is used, report the As#2 result (2nd As value listed) which has an ^{78}Se correction equation.

15.3.3. V#1 (1st V value listed) has a ^{52}Cr and ^{53}Cr correction equation. V#2 (2nd V value listed) has no corrections; this is for informational purposes only.

15.3.4. Highlight the raw data for the acceptable values of the requested elements. If an isotope other than the first one listed is chosen, make a dash with highlighter pen into the margin to the left of isotope.

15.3.5. For indications of possible interference, refer to the interference information in the Equations window of the method.

15.4. QC Samples Review

15.4.1. Method QC Solutions:

15.4.1.1. QCS analysis should be performed when new standards are prepared to verify the accuracy of the calibration standards.

15.4.1.1.1. Whenever the QCS is analyzed the results should be within the certified QC range. If the limits are not met, rerun the QCS. If the limits are still not met, re-preparation of standards and/or recalibration may be indicated. Notify the supervisor of QCS problems.

15.4.1.2. Calibration Verifications (ICV, CCV): Calibration verification QC samples are run to verify calibration stability. The ICV is run immediately after calibration and the CCV is run before and after groups of up to 10 samples. Both ICV and CCV readings should be ±10% of the true value for all requested elements for each isotope used. If this limit is not met, recalibrate and rerun the samples; or rinsing (10-15 minutes) may correct a matrix carry-over effect, followed by rerunning the CCV, CCB, and the affected samples.

15.4.1.3. Calibration Blanks (ICB, CCB): Calibration blanks are run after every ICV or CCV to verify baseline stability and to check for carry-over. Both ICB and CCB readings should be <RL (DOD projects require that no analyte is detected >2 x MDL). If this limit is not met, recalibrate the blank, rerun the CCV, CCB, and then rerun the samples that were not bracketed by in-control blanks. Rinsing (5-10 minutes) before recalibrating the blank may correct a carry-over effect.

15.4.1.4. Interference Check Solutions: The interference check solutions, ICSA and ICSAB, are run near the start of the run or once every 12 hours, whichever is more frequent, to verify that the interference correction equations are adequate to correct for some common interferents. See Appendix 8 for the concentrations of these solutions.

15.4.1.4.1. ICSA: The ICSA contains the following interferents: Al, C, Ca, Cl, Fe, K, Mg, Mo, Na, P, S, and Ti. The interferents which are analyzed should be ±20% of true values. The analyte (analytes which are spiked in the ICSAB) concentrations should typically run ±2RL, although no QC limits are used. DOD requires the absolute value of all non-spiked analytes to be <2 X MDL (unless they are a verified trace impurity from one of the spiked analytes.

15.4.1.4.2. ICSAB: The ICSAB solution contains the same interferents as the ICSA at the same levels, plus some commonly requested analytes. These analytes are Ag, As, Cd, Co, Cr, Cu, Mn, Ni, and Zn; they are all at 20 µg/L in the ICSAB solution. Both the interferents which are analyzed and the analytes should be ±20% of true values.

15.4.1.5. Low Check Standard: This low level standard verifies the accuracy of the instrument

at the RL for all analytes. The limits are ±1/2 RL. DOD requires limits within 20% of the expected value. If the concentrations are outside this range, the analyst should look for the possible cause (e.g. calibration blank intensities too high, baseline drift, contamination, etc.). Re-preparation of the blank, recalibration of the blank, or rinsing the instrument may be required.

15.4.1.6. Linear Range Solutions: The linear range of an instrument for an isotope is the upper limit of accurate quantitation with practical rinse-down times. It varies for each isotope and with instrumental conditions. Two LR solutions can be analyzed on a daily basis if the samples are expected or found to be above the calibration range. LR200 (200 µg/L non-minerals, 20,000 µg/L minerals) and LR300 (300 µg/L non-minerals, 30,000 µg/L minerals) are the linear range solutions. The LR solution concentration must be within 10% of the true value to extend the calibration range of that isotope. DOD requires samples to be diluted and reanalyzed (if possible) to bring them within the calibration curve.

15.4.2. Digestion / Batch QC Samples

15.4.2.1.1. Method Blank (MB): A method blank (MB) should be run with every client batch of samples or every CLP sample delivery group (SDG). A minimum of one MB must be run for every batch of 20 samples of the same matrix.

15.4.2.1.2. MB values greater than the RL indicate laboratory or reagent contamination.

15.4.2.1.3. The method blank should be <1/2RL, unless the element is a common laboratory contaminant when it should be <RL. If the requested element is detected in the method blank ≥RL, then all the associated samples may need to be re-digested and reanalyzed unless all samples are > 10 times the detected method blank concentration. The analyst should fill out a corrective action form and the supervisor should be informed.

15.4.2.2. Laboratory Control Sample, Reference Sample or Method Blank Spike (LCS / REF / MBSPK)

15.4.2.2.1. One LCS, REF or MBSPK (of the same matrix type as the samples), should be analyzed with each batch of samples of the same preparation procedure.

15.4.2.2.2. For an aqueous LCS, a REF sample or an MBSPK must be carried through all the procedures with which the samples are subjected. The MBSPK is prepared by spiking an aliquot of the method blank at the appropriate levels. The REF sample is prepared by diluting a QC standard to the appropriate levels.

15.4.2.2.3. The percent recovery for an aqueous LCS is calculated according to the

following:

$$\%R = \frac{LCS}{s} * 100$$

Where:

%R: Percent Recovery

LCS: LCS, REF, or MBSPK Results

s: True concentration

15.4.2.2.4. The Percent Recovery for the aqueous LCS, REF sample, or MBSPK should be within the required control limits of 80-120% (85-115% for Method 200.8). If the recovery is outside the QC limits, then the source of the problem shall be identified and resolved before re-preparation of the sample batch. The analyst should fill out a corrective action form, and the supervisor should be informed.

15.4.2.2.5. For a soil reference, typically an ERA SRM, the certified ranges are used as recovery limits, though a client may specify other statistical limits. The reference sample concentration must be calculated in mg/kg units. If the recovery is outside the certified range, then the source of the problem shall be identified and resolved before re-preparation of the sample batch. The analyst should fill out a corrective action form, and the supervisor should be informed.

15.4.2.3. Matrix Spike/Matrix Spike Duplicate.

15.4.2.3.1. . A matrix spike should be run with every client batch of samples or every CLP sample delivery group (SDG). The laboratory must spike a known amount of analyte into a minimum of one sample per batch not to exceed 20 samples. DOD requires an MS/MSD for each batch of 20 samples.

15.4.2.3.2. Calculate the percent recovery for the matrix spike as follows:

$$\%R = \frac{C_s - C}{s} * 100$$

Where:

%R: Percent Recovery

Cs : Measured concentration in fortified sample matrix

C: Measured concentration in unfortified sample

s: Amount of analyte added to sample matrix

15.4.2.3.3. The recovery of the matrix spike should be within the designated QC limits of 75-125%. If it is not within this range, and the LCS recovery is acceptable, the data user

will be informed that the result in the unfortified sample is suspect due to heterogeneity or an uncorrected interference effect. Recovery calculations are not required if the concentration of the analyte added is <25% of the analyte present in the sample. If the matrix spike %R is outside the QC limits, the analyst should fill out a corrective action form and the supervisor should be informed. For DOD spike recovery acceptance criteria is 85-115%.

15.4.2.4. Laboratory Duplicate

15.4.2.4.1. A laboratory duplicate should be run with every client batch of samples or every CLP sample delivery group (SDG). There must be at least one laboratory duplicate prepared with each batch of samples not to exceed 20 samples.

15.4.2.4.2. Calculate the RPD as follows:

$$RPD = \frac{D_1 - D_2}{(D_1 + D_2)/2} * 100$$

Where:

RPD: Relative Percent Difference

D1: Measured concentration of first sample

D2: Measured concentration of replicate sample

15.4.2.4.3. A control limit of ≤20 % shall be used for the RPD if the sample concentration is ≥5RL. A control limit of ±1RL shall be used if either the sample or the duplicate sample concentration is <5RL. If it is not within this range, the analyst should fill out a corrective action form and the supervisor should be informed. The project manager will be informed of the poor duplication, and project specific corrective action will be taken.

16. **Contingencies for Handling Out-of-Control or Unacceptable Data**

16.1. Calibration: If the calibration does not meet the criteria in Section 11.3.3.2, then corrective action shall be taken before proceeding with recalibration. This could involve uptake rate optimization, clog removal, re-preparation of calibration standards, etc.

16.2. Instrumental QC checks: If an instrumental QC sample (CV, CB, QCS, etc.) is out of control, then corrective action shall be taken before proceeding with analysis. This could involve recalibration of the blank (resetting the baseline), re-preparation of calibration standards and recalibration, analysis of an alternate QCS, etc.

16.3. Instrument malfunctions: When instrument malfunctions occur, consult with other experienced

ICP-MS operators or the supervisor for guidance. The maintenance logbook, the ICP-MS hardware manual, or the ICP-MS software manual could be helpful for troubleshooting.

16.4. In the event of significant QC failure, analysis will stop and the analyst will perform corrective action as discussed above. In general, out-of-control sample results will not be reported. Reruns will be conducted based on sample availability. If insufficient sample remains the client will be notified to determine an appropriate course of action.

17. Waste Management

17.1. Metals analysis results in the generation of two waste streams which must be given special treatment.

17.1.1. Acidic solutions having pH <2 and little or no trace metal concentrations: These should be neutralized to pH 7 and then sink discharged. A log book for Elementary Neutralization Activities is available in the Metals Instrument Lab. The Date, Source, Volume, Initial and Final pH, and Analyst initials should be recorded each time waste is neutralized.

17.1.2. Samples and sample preparation solutions having pH <2 and Hazardous levels of trace metals. A list of such samples is computer generated from sample analysis data. This list is used to mark all samples and sample solutions requiring segregation and disposal as Hazardous Waste. All such wastes are collected in a polyethylene satellite container in the Metals Instrument Lab or in the Metals Waste Drum in the Hazardous Waste Accumulation site. When the containers are full the Hazardous Materials Coordinator is notified for off site disposal.

18. Method References

18.1. "Methods for the Determination of Metals in Environmental Samples - Supplement 1", "Method 200.8 Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma – Mass Spectrometry", Revision 5.4, EPA-600/R-94-111, May 1994.

18.2. ELAN® 6000 Hardware Manual, 1995, Perkin-Elmer Corporation

18.3. ELAN® 6000 ICP-MS Software Manual, 1995, Perkin-Elmer Corporation

18.4. EPA SW-846 "Method 6020 Inductively Coupled Plasma – Mass Spectrometry", Revision 0, Sept 1994

19. Appendices

19.1. Appendix 1: Internal Standards and Tuning Solutions

19.2. Appendix 2: Calibration Stocks

19.3. Appendix 3: Calibration Intermediate

19.4. Appendix 4: Calibration Standards and Linear Range Solutions

19.5. Appendix 5: Inorganic Ventures ICV

19.6. Appendix 6: Dual Detector Solution

19.7. Appendix 7: Low Check Solution

19.8. Appendix 8: Interference Check Solutions (ICSA, ICSAB)

19.9. Appendix 9: Interference Correction Equations

19.10. Appendix 10: Analytical Isotopes and Additional Monitored Isotopes

19.11. Appendix 11: ICP-MS Reporting Limits

19.12. Appendix 12: Troubleshooting

19.13. Appendix 13: Instrument Maintenance

Appendix 1

ICP-MS INTERNAL STANDARDS AND TUNING SOLUTIONS

All concentrations in mg/L

1. INTERNAL STANDARD SOLUTION

Use at 1/100 for final levels, all concentrations in mg/L

ELEMENT	STOCK CONC	VOL OF STK IN 1000 ml	INT CONC	FINAL CONC in 1% HNO_3
^{6}Li	1000	30.0	30.0	0.300
Sc	1000	2.0	2.0	0.020
Ge	1000	10.0	10.0	0.100
Y	1000	1.0	1.0	0.010
In	1000	1.0	1.0	0.010
Tb	1000	1.0	1.0	0.010
Bi	1000	1.0	1.0	0.010

2. TUNING SOLUTION

Use at 1/100 for final levels, all concentrations in mg/L

ELEMENT	STOCK CONC	VOL OF STK IN 100 ml	INT CONC	FINAL CONC in 1% HNO_3
Be	1000	0.1	1.0	0.010
Mg	1000	0.1	1.0	0.010
Co	1000	0.1	1.0	0.010
In	1000	0.1	1.0	0.010
Ba	1000	0.1	1.0	0.010
Ce	1000	0.1	1.0	0.010
Pb	1000	0.1	1.0	0.010
Ba	1000	0.1	1.0	0.010

Appendix 2

ICP-MS CALIBRATION STOCKS

STOCK #1

Prepare in 1% trace grade HNO_3. All concentrations in mg/L

ELEMENT	STOCK CONC	VOL OF STD IN 100 mL	STOCK CONC
Ag	10000	1.0	100
As	10000	1.0	100
Ba	10000	1.0	100
Be	10000	1.0	100
Cd	10000	1.0	100
Co	10000	1.0	100
Cr	10000	1.0	100
Cu	10000	1.0	100
Mn	10000	1.0	100
Ni	10000	1.0	100
Pb	10000	1.0	100
Se	10000	1.0	100
Tl	10000	1.0	100
Th	10000	1.0	100
U	10000	1.0	100
V	10000	1.0	100
Zn	10000	1.0	100

STOCK #2

Prepare in DI H_2O

Sb	10000	1.0	100
Mo	10000	1.0	100

Appendix 3

ICP-MS CALIBRATION INTERMEDIATE

All concentrations in mg/L
Prepare in 1% trace grade HNO_3

ELEMENT	STOCK CONC	VOL OF STK **IN 100 mL**	INT CONC
Al	10000	10.0	1000
Ca	10000	10.0	1000
Fe	10000	10.0	1000
K	10000	10.0	1000
Mg	10000	10.0	1000
Na	10000	10.0	1000
STOCK #1	see table 2	10.0	10
STOCK #2	see table 2	10.0	10

Appendix 4

ICP-MS CALIBRATION STANDARDS and LINEAR RANGE SOLUTIONS

Add the intermediate to a 100 mL volumetric flask containing 1.0 mL trace metal grade HNO_3 and bring to volume. Standard 3 is made up to 200 mL with addition of 2.0 mL HNO_3.

Standard	mL Intermediate	Concentration µg/L in 1% HNO_3	
		Non-minerals	Minerals
1	0.1	10	1000
2	0.2	20	2000
3	1.0	50	5000
4	1.0	100	10000
LR200	2.0	200	20000
LR300	3.0	300	30000

Appendix 5

ICP-MS INORGANIC VENTURES ICV

All concentrations in mg/L

ELEMENT	STOCK CONC	FINAL CONC
Ag	2.5	0.050
Al	250	5.000
As	2.5	0.050
Ba	2.5	0.050
Be	2.5	0.050
Ca	250	5.000
Cd	2.5	0.050
Co	2.5	0.050
Cr	2.5	0.050
Cu	2.5	0.050
Fe	250	5.000
K	250	5.000
Mg	250	5.000
Mn	2.5	0.050
Mo	2.5	0.050
Na	250	5.000
Ni	2.5	0.050
Pb	2.5	0.050
Sb	2.5	0.050
Se	4.0	0.080
Th	2.5	0.050
Tl	2.5	0.050
U	2.5	0.050
V	2.5	0.050
Zn	2.5	0.050

Appendix 6

ICP-MS DUAL DETECTOR CALIBRATION SOLUTION

All concentrations in mg/L
Use Intermediate at 1/10

ELEMENT	STOCK CONC	VOL OF STOCK IN 200 ml	INTERMED CONC	FINAL CALIB SOL'N CONC µg/L (in 1% HNO_3)
Ag	1000	0.5	2.5	0.25
Al	1000	0.5	2.5	0.25
As	1000	0.5	2.5	0.25
Ba	1000	0.5	2.5	0.25
Be	1000	0.5	2.5	0.25
Bi	1000	0.5	2.5	0.25
Ca	1000	0.5	2.5	0.25
Cd	1000	0.5	2.5	0.25
Co	1000	0.5	2.5	0.25
Cr	1000	0.5	2.5	0.25
Cu	1000	0.5	2.5	0.25
Fe	1000	0.5	2.5	0.25
Ge	1000	0.5	2.5	0.25
In	1000	0.5	2.5	0.25
K	1000	0.5	2.5	0.25
^{6}Li	1000	0.5	2.5	0.25
Mg	1000	0.5	2.5	0.25
Mn	1000	0.5	2.5	0.25
Mo	1000	0.5	2.5	0.25
Na	1000	0.5	2.5	0.25
Ni	1000	0.5	2.5	0.25
Pb	1000	0.5	2.5	0.25
Sb	1000	0.5	2.5	0.25
Sc	1000	0.5	2.5	0.25
Tb	1000	0.5	2.5	0.25
Th	1000	0.5	2.5	0.25
Tl	1000	0.5	2.5	0.25
U	1000	0.5	2.5	0.25
V	1000	0.5	2.5	0.25
Y	1000	0.5	2.5	0.25
Zn	1000	0.5	2.5	0.25

Appendix 7

ICP-MS LOW CHECK SOLUTION

Use Intermediate at 0.05/100 for final levels

ELEMENT	STOCK CONC mg/L	VOL OF STOCK IN 100 mL	INT CONC mg/L	FINAL CONC µg/L (in 1% HNO_3)
Ag	1000	0.04	0.4	0.2
Al	10000	0.4	40	20
As	1000	0.04	0.4	0.2
Ba	1000	0.1	1	0.5
Be	1000	0.04	0.4	0.2
Ca	10000	1	100	50
Cd	1000	0.04	0.4	0.2
Co	1000	0.04	0.4	0.2
Cr	1000	0.1	1	0.5
Cu	1000	0.1	1	0.5
Fe	10000	0.4	40	20
K	10000	0.4	40	20
Mg	10000	0.4	40	20
Mn	1000	0.1	1	0.5
Mo	1000	0.04	0.4	0.2
Na	10000	2	200	100
Ni	1000	0.1	1	0.5
Pb	1000	0.2	2	1
Sb	1000	0.04	0.4	0.2
Se	1000	0.1	1	0.5
Th	1000	0.04	0.4	0.2
Tl	1000	0.04	0.4	0.2
U	1000	0.04	0.4	0.2
V	1000	0.04	0.4	0.2
Zn	1000	0.8	8	4

Appendix 8

ICP-MS Interference Check Solutions: ICSA and ICSAB
All concentrations in mg/L
* AR-6020ICS-A10 Custom Stock Solution from Inorganic Ventures.

ELEMENT	ICSA STOCK CONC*	ICSA FINAL CONC	ICSAB STOCK CONC	ICSAB FINAL CONC (in 1% HNO_3)
Ag			2.0	0.02
Al	1000	20		20
As			2.0	0.02
C	2000	40		40
Ca	1000	20		20
Cd			2.0	0.02
Cl	10000	200		200
Co			2.0	0.02
Cr			2.0	0.02
Cu			2.0	0.02
Fe	1000	20		20
K	1000	20		20
Mg	1000	20		20
Mn			2.0	0.02
Mo	20	0.4		0.4
Na	1000	20		20
Ni			2.0	0.02
P	1000	20		20
S	1000	20		20
Ti	20	0.4		0.4
Zn			2.0	0.02

Appendix 9

ICP-MS INTERFERENCE CORRECTION EQUATIONS

ANALYTE	MASS	EQUATION
V	50.944	-3.127*(Cr53-0.113*Cr52)
Fe	53.94	-0.028226*Cr52
As #1	74.922	-3.127*(ArCl 77-(0.815*Se82))
As #2	74.922	-3.127*(ArCl 77-(0.3209*Se78))
Se	77.917	-0.030435*Kr83
Se	81.917	-1.008696*Kr83
Mo	97.906	-0.110588*Ru101
Cd	110.904	-1.073*(MoO 108-(0.712*Pd106))
Cd	113.904	-0.026826*Sn118
In	114.904	-0.014032*Sn118
Sb	122.904	-0.127189*Te125
Pb	207.977	+1*Pb206+1*Pb207

Appendix 10

ANALYTICAL ISOTOPES AND ADDITIONAL MONITORED ISOTOPES

1. ANALYTICAL ISOTOPES		2. ADDITIONAL ISOTOPES	
ELEMENT	ISOTOPE (S)	ELEMENT	ISOTOPE (S)
Li	6	Ru	101
Be	9	Pd	106
Sc	45	Sn	118
Na	23	Te	125
Mg	24		
Al	27		
K	39		
Ca	44		
V	51		
Cr	52, 53		
Fe	54, 57		
Mn	55		
Co	59		
Ge	72		
Ni	60, 62		
Cu	63, 65		
Zn	66, 67, 68		
As	75		
Se	78, 82		
Mo	98		
Y	89		
Kr	83		
Ag	107		
Cd	111, 114		
In	115		
Sb	121, 123		
Ba	135, 137		
Tb	159		
Tl	205		
Pb	206, 207, 208		
Bi	209		
Th	232		
U	238		

Appendix 11

ICP-MS REPORTING LIMITS

ELEMENT	RL µg/L
Ag	0.2
Al	20
As	0.2
Ba	0.5
Be	0.2
Ca	50
Cd	0.2
Co	0.2
Cr	0.5
Cu	0.5
Fe	20
K	20
Mg	20
Mn	0.5
Mo	0.2
Na	100
Ni	0.5
Pb	1
Sb	0.2
Se	0.5
Th	0.2
Tl	0.2
U	0.2
V	0.2
Zn	4

Appendix 12

Troubleshooting

1. **The following sections describe some commonly occurring problems and proposed solutions:**

1.1. Poor Curve Fit

1.1.1. Poor curve fit may require individual standards to be rerun or re-prepared; a complete recalibration may be required.

1.1.2. If the curve fit appears to be off between pulse readings (less than approx. 1.5 million-cps) and analog readings (approx. 2 million to 1 billion cps), then a new dual detector optimization/calibration may be required. Poor Pb or Tl curve fit is a good indicator of when this is necessary (other indicator elements are Mn, Th, U).

1.2. Dual Detector Calibration/Optimization (enter the settings into the maintenance log)

1.2.1. Copy **arioptimize.dac** on the C drive the sub-directory Elandata\Optimize to the "YYYY"\Optimze folder and rename as dualyymmdd.dac using the date of the last dual detector calibration (where yy are the last 2 digits of the year, mm is the month and dd is the day of the month).

1.2.2. Perform a pulse stage detector optimization (see pages 3-19 to 3-20 of the ELAN® software guide): Aspirate Standard 1 (10 µg/L non-minerals and 1000 µg/L minerals). Open **ARIoptimize.wrk** workspace, highlight Pulse Stage Voltage, and click on Get Analyte List (Ge), click on Optimize. After the standard has run, review the graph on the Interactive page. The curve should be just bracketed by the graph limits if the correct range has been set. The pulse optimum voltage will be automatically set by the software: marked by an open square on the graph. Compare this to the graph on page 3-20 of the ELAN® software guide. If the optimum is set too low or too high, click on the appropriate point on the graph. Print the graph and save the file.

1.2.3. Perform an analog stage detector optimization (see pages 3-21 to 3-23 of the ELAN® software guide): aspirate the dual detector calibration solution. Open arianalog.wrk workspace, highlight Analog Stage Voltage, and click on Get Analyte List (Mg) and click on Optimize. After the optimization is complete, write down the current value (analog optimum voltage), the target gain and the achieved gain. Save the file.

1.2.4. Perform another pulse stage optimization (see section 1.2.2).

1.2.5. Perform a dead time correction (see pages 3-24 to 3-25 of the ELAN® software guide): aspirate the dual detector calibration solution. Open "arideadtime.wrk" workspace. Click on Get Analyte List (Ge) and click on Calibrate. After the standard has run, from the main window menu choose Options, then Configurations, set the dead time (ns) to 35, click on OK, click on Exit. In the Dataset window highlight the last 2 sample rows, click on Clear Calibration, click on Get Analyte List and click on Calibrate from Dataset. Make a table of ns setting and r-values. Change the dead time 5 ns higher, and then repeat the above procedure to calculate the r-value. Record the r-value at each setting (ns). Repeat this process up to 90 ns. From the list of ns and r-values, choose the highest r-value less than 75ns. The associated dead time setting (ns) should be entered into configurations/deadtime. Save the "arioptimize.dac" file. To store this dead time value, exit the ELAN® software before taking other measurements. Restart the ELAN® Software and restart the peristaltic pump.

1.2.6. Perform a dual detector calibration (See page 3-26 of the ELAN® software guide): Aspirate the dual detector calibration solution. Open "aridualdetcal.wrk" workspace, click on Clear Calibration, click on Get Analyte List, click on Calibrate. This calibration will take approximately 10 minutes to run. Save the "arioptimize.dac" file. Print an optimization summary. Note the range of gain values from the optimization print out; record the range of gain values in the maintenance log and compare with those from several previous dual detector calibrations. On the optimization summary, make a note of any r-values <0.9995 and number of points <10. On the Interactive page, check the individual calibration graphs for good curve fits.

1.2.7. Run a daily performance check to check sensitivity. Compare the sensitivity before and after the dual detector calibration.

1.3. Poor relative standard deviation (precision) on standards and samples: Poor RSDs have many potential causes. Recalibrate if any adjustments are made.

1.3.1. First, check that the peristaltic pump tubing is in good condition and not worn. When the autosampler probe is removed and reinserted in the rinse solution an air bubble will be visible in the tubing. Watch the progress of this bubble and check that the flow is smooth without any pulsation. Only adjust the tension on the pump tubing beds if necessary.

1.3.2. Check that the nebulizer is operating properly by first measuring the sample uptake rate. It should be 1.1 to 1.2 mL/min. If necessary, the aerosol may be checked with the plasma off and the spray chamber removed. Turn on the nebulizer gas and the peristaltic pump; there should be a visible aerosol leaving the spray chamber. If there is not, clean or replace the

nebulizer.

1.3.3. Check that the interface cones are in good condition and the orifices of both cones are round and of the proper size.

1.4. Low Sensitivity

1.4.1. First check the x-y adjustment of the torch to sampler cone (see pg. 3-9 of the software guide).

1.4.2. Check the sample uptake rate as recommended in section 1.3.2. If it is too low, then check the tubing for clogs and check the air bubble progress in the uptake tubing.

1.4.3. With the plasma off, check the sampler cone to torch spacing using the Perkin Elmer spacer tool (see pg. 5-16 of the ELAN® Hardware Guide). Also check the condition of the cones.

Appendix 13

Instrument Maintenance

1. **Daily Maintenance**

1.1. Cones: The sample cone is inspected daily for build-up of salts and soot. No cleaning is required for light build-up. Swab lightly with a slightly DI water moistened cotton swab to remove moderate build-up. Allow to air dry for 5 minutes. If the salt build-up is heavy, remove both the sample and the skimmer cones, and sonicate after the removal of their O-rings (see page 5-27 of the ELAN® Hardware Guide). Sonicate in 1% Citrinox solution 20 minutes, first ensuring that air bubbles are eliminated from inside the cones; use the cleaning beaker specified for the skimmer cone. Rinse well with DI water, then thoroughly air dry before using or storing. Perform an x-y adjustment after the cones are replaced (see page 3-9 of the software guide).

1.1.1. Peristaltic Pump Tubing: Inspect the peristaltic pump tubing daily for flat areas and wear. Typically they require replacement after 3 to 5 sample runs.

1.1.2. Water Chiller: Check the water chiller daily for the following settings. The temperature meter should be 17-19°° C. The temperature can be adjusted using the knob below the meter (wait 5 minutes between adjustments). Check the coolant level under the small square panel on the top right corner of the chiller. It should be almost full (to the line below the cap threads). Otherwise, fill with DI water to the line. Check the pressure gauge on the front panel; it should be 55-56 psi. The pressure can be adjusted at the regulator located on the back (see page 5-31 of the hardware guide).

1.1.3. Vacuum Pumps: Check the oil level windows daily on both roughing vacuum pumps (located inside the front panel) for adequate oil and for an oil color change. The interface vacuum pump located on the right requires oil changes more frequently; watch for the color of the oil to change. Darkening of the oil color indicates an oil change is required (see page 5-35 of the hardware guide).

1.2. Maintenance as Needed

1.2.1. Nebulizer: The gemtip crossflow nebulizer may require maintenance if a lowered uptake rate is measured (< 1.0 mL/min). Check all tubing connectors for clogs first. To change the gemtips, extinguish the plasma, and replace the gemtips with the spare ones. Note that the argon tip has a red tip and the sample tip has a clear tip. If the uptake rate is still low, extinguish the plasma, remove the nebulizer and check the aerosol production.

1.2.2. Torch: The torch, which is made up of a quartz torch body, a ceramic injector and a lexan adapter/base, requires replacement periodically. Torch body discoloration is acceptable unless performance is affected. If the torch has an arc spot, be aware that it may develop into a crack or hole. Sensitivity losses may necessitate torch maintenance to eliminate it as a possible cause.

1.2.3. Load Coil: The load coil requires replacement periodically. Inconsistent torch lighting may necessitate cleaning of the coil. Replace the coil if the surface becomes pitted from excessive arcing.

1.2.4. Ion Lens Cleaning: This is required periodically; about every 3 months, depending on the volume and the types of samples analyzed. It may be necessitated by AutoLens® irregularly shaped peaks, by low daily performance Mg intensity (representative of low masses), or by CVs repeatedly out of control for only low masses or high masses.

1.2.4.1. On the Instrument page, click on Vacuum STOP: The plasma should already be off. Wait about 5 minutes for the vacuum to dissipate.

1.2.4.2. Open the instrument cover and loosen the 6 screws on the top of the vacuum chamber. Lift the top off the vacuum chamber. Wear powder-free gloves when working in the vacuum chamber.

1.2.4.3. In the right chamber, disconnect the gray leads from each other. Loosen the plastic set screw (gray lead attached) on the lens cage bracket. Slide the lens to the left. Loosen the retaining ring which holds the lens cage bracket in place. Remove the lens assembly without disturbing the aperture plate which is held in place by the lens cage bracket.

1.2.4.4. Replace the lens with a spare pre-cleaned one. The lens is cleaned by sonicating it in Methanol for 15 minutes, rinsing thoroughly with deionized water, and thoroughly drying. Store the lens in a Kimwipe® wrapper labeled with the number of uses. A lens will usually last 3 uses.

1.2.4.5. Without disturbing the aperture plate, place the lens assembly into place and tighten the retaining ring. Before tightening the plastic set screw, slide the lens to the right and stop 1-2 mm from the photon stop. Tighten the plastic set screw. Reconnect the gray leads to each other.

1.2.4.6. Replace the vacuum chamber cover and tighten the 6 screws. On the Instrument page, click on Vacuum START. The vacuum pumps may take about 2-3 hours to pump down to working vacuum. After the working vacuum has been achieved, retighten the 6 screws on the vacuum chamber cover.

Analytical Resources, Incorporated
Analytical Chemists and Consultants

Standard Operating Procedure

Volatile Organics Analysis
(Gas Chromatography/Mass Spectrometry)

SOP 700S
Version 013

Revision Date: 01/13/10
Effective Date: 01/13/10

Prepared By:

Aron Rigg, Patrick Basilio, Jianqing Zhou, Van Spohn

Approvals:

Laboratory / Section Manager

Quality Assurance

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Annual Review

SOP Number: 700S

Title: Volatile Organics Analysis (Gas Chromatography/Mass Spectrometry)

The ARI employee named below certifies that this SOP is accurate, complete and requires no revisions

Name	Reviewer's Signature	Date

1. Scope and Application

1.1. This document describes the procedures for performing volatile organic analyses (VOA) used by Analytical Resources Inc. (ARI). The procedures are based on EPA Method 8260C and EPA Method 8000C Revision 3, March, 2003 referenced in Section 19.1. Some text is directly from those documents. The procedure will identify and quantify volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. Water-soluble volatile compounds may be included in this analytical technique, however, for the more soluble compounds, the quantitation limits are approximately ten times higher because of poor purging efficiency. ARI's routine target analytes for this procedure are listed in Appendix 20.3. Samples may be analyzed for additional analytes on a project specific basis.

1.2. This method is applicable to most environmental sample matrices, including aqueous (ground water, surface water, waste water, TCLP extracts), solid (soil, sediment, sludge), air samples in Tedlar bags and waste (waste solvent, oily waste, mousse, tar, polymeric emulsion, filter cake, spent carbon, etc) samples.

1.3. ARI uses several sample preparation techniques to achieve project and/or client specific detection limits. Routine techniques are summarized in Table 01.

Table 01

Typical VOA Analyzes performed by ARI

Sample Matrix	Sample size	Extraction Technique	Estimated LOQ
Water	5 mL	Direct Purge & Trap	1 – 5 µg/L
Water	10 mL	Direct Purge & Trap	0.2 – 5 µg/L
Soil / Sediment	5 g	Direct Purge & Trap	1.0 – 5.0 µg/kg
Medium Level Solids	5 g	Methanol Extraction	50 – 250 µg/kg

1.4. ARI routinely performs method detection limit (MDL) studies for each extraction and analytical method performed using this SOP. The results of these studies help define the reporting limits of data generated. The results are kept by the QA department, and are distributed to the bench chemists and the LIMS administrator. Current MDLs are published in ARI's Laboratory Quality Assurance Plan (LQAP).

1.5. This document describes a purge-and-trap, gas chromatographic/mass spectrometric (GC/MS) procedure. This method is restricted to use by, or under the supervision of, analysts experienced in the use of purge-and-trap systems and gas chromatograph/mass spectrometers, and skilled in the interpretation of mass spectra and their use as a quantitative tool.

2. Summary of Procedure

2.1. This method requires separate preparation and analysis procedures. Sample preparation is outlined in Section 11.5. Analysis of prepared sample is accomplished using a Purge & Trap GC-MS instrument described in Section 11.3. Software in the GC-MS system automates the data acquisition and reduction process. Most calculations described in this document are performed automatically by the Target™ Software running on the Target Server. The formulae are provided for reference and may be used to manually verify calculated results.

2.2. There are two basic sample preparations.

- 2.2.1. Direct purge and trap where the sample is analyzed with no initial extraction or preparation.
- 2.2.2. Methanol extraction is used to dissolve volatile organic compounds in Methanol which is diluted and analyzed.

2.3. Following sample preparation, volatile organic compounds are automatically purged from the sample and injected into the gas chromatograph using a purge-and-trap equipped GC-MS system.

- 2.3.1. Compounds purged from the sample using Helium are trapped in a tube containing suitable sorbent materials.
- 2.3.2. The sorbent tube is heated and back flushed with helium to desorb trapped sample components directly onto the GC-MS system following a 1:40 split.

2.4. A narrow bore capillary GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS).

2.5. Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing resultant GC retention times and mass spectra.

2.6. Identified target analytes are quantified by comparing the detector responses of each analytes' characteristic mass ion and internal standard characteristic mass ion to the responses of these ions in a calibration curve prepared using analytes at known concentration.

2.7. Detection limits for all analytes quantitated using this SOP are set using the low point of the initial calibration curve and validated by method detection limit studies.

- 2.7.1. MDL studies are performed each year for each analyte by each preparatory and analytical method.
- 2.7.2. MDL and reporting limit (RL) values may be found for each analyte in the ARI LQAP.

3. Definitions

3.1. CALGAS: perfluoro-tri-n-butylamine (FC-43), CAS 311-89-7.

3.2. Continuing Calibration Verification (CCV): a process used to verify that the current instrument calibration is acceptable.

3.3. Continuing Calibration Verification Standard (CCVS): A standard prepared at the mid-point concentration of the initial calibration, and prepared from the same source as the initial calibration.

3.4. EICP- Extracted Ion Current Profile- A plot of the abundance of a specific ion as a function of time.

3.5. Holding Blank: A blank water sample stored along with client samples. A holding blank is analyzed periodically to determine if there is cross contamination of samples introduced during storage in the laboratory. See Section 16 for contamination of holding blanks.

3.6. Initial Calibration (ICAL): a minimum of 5 points, with 6 points typical.

3.7. Initial Calibration Verification (ICV): a process used to verify that the current instrument calibration is acceptable.

3.8. Initial Calibration Verification Standard (ICVS): A mid point concentration standard from a source different than that used for the initial calibration used to demonstrate the validity of the initial calibration. The ICVS is equivalent to the Second Source Standard. The second source standard must be purchased from a different manufacturer than the calibration standard whenever possible.

3.9. Instrument Blank (IB): A QC sample made by adding surrogates to organic free water (OFW) used to measure instrument background during an analytical run.

3.10. Internal Standard (IS): internal standards are compounds added to each standard, sample, and QC sample such that their concentration is the same in each of these sample types. Target analyte response is normalized to the response of an internal standard.

3.11. Laboratory Control Sample (LCS): OFW spiked with verified amounts of analytes. It is generally used to establish intra-laboratory or analyst-specific precision or to assess the performance of all or a portion of the measurement system.

3.12. Laboratory Control Sample Duplicate (LCSD): A replicate LCS often used to assess the precision of an analytical method. When insufficient sample volumes exist to perform a required MS/MSD analysis, an LCS/LCSD may be performed to assess the precision of the analytical method. The LCSD is prepared and analyzed identically to the LCS. ARI fortifies the LCS/LCSD with all target analytes.

3.13. LIMS (Laboratory Information Management System): Software used to compile and report final chromatographic data.

3.14. Limit of Detection (LOD): The MDL determined following the procedure in 40 CFR 136, Appendix B.

3.15. Limit of Quantitation (LOQ): A concentration equivalent to the low point on an instrument calibration curve.

3.16. Matrix Spike (MS): A sample prepared by adding a known mass of target analyte(s) to a specified amount of sample matrix for which an independent estimate of target analyte concentration is available. Matrix spikes are used to determine the effect of the sample matrix on the recovery efficiency of an analytical method. ARI fortifies Matrix Spike samples with all target analytes.

3.17. Matrix Spike Duplicate (MSD): A replicate matrix spike prepared in the laboratory and analyzed to obtain a measure analytical precision.

3.18. Method Blank (MB): A sample of OFW, free of any analytes of interest that is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures.

3.19. Organic Free Water (OFW): ASTM Type 1 water produced by ARI's centralized water purification system and run through a bed of activated charcoal in the VOA laboratory.

3.20. RIC- Reconstructed Ion Current- A plot of the total instrument response versus time

3.21. RRT-Relative Retention Time- the elution time of an analyte relative to the elution time of its associated internal standard

3.22. Scan Descriptor: Defines a specific mass range for analytes of interest.

3.23. Second Source Standard: A standard from a different manufacturer or lot number other than the standard used to calibrate an instrument. The SSS is used to prepare the Initial Calibration Verification Standard.

3.24. Selective Ion Monitoring (SIM) - a detection method wherein the mass spectrometer is programmed only to scan for certain masses instead of a broad range of masses. See SOP 703S

3.25. Solvent Blank – A clean sample (OFW and/or MeOH) analyzed using the same conditions as a regular sample. A solvent blank detects system contamination and assures the purity of the solvent. Used as a MB for MeOH extracted samples.

3.26. Surrogate – A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

3.27. Target™ - A chromatography software package. ARI uses Target™ software to identify and quantify GC and GC-MS target analytes

4. Interferences

4.1. Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be necessary.

3.16. Matrix Spike (MS): A sample prepared by adding a known mass of target analyte(s) to a specified amount of sample matrix for which an independent estimate of target analyte concentration is available. Matrix spikes are used to determine the effect of the sample matrix on the recovery efficiency of an analytical method. ARI fortifies Matrix Spike samples with all target analytes.

3.17. Matrix Spike Duplicate (MSD): A replicate matrix spike prepared in the laboratory and analyzed to obtain a measure analytical precision.

3.18. Method Blank (MB): A sample of OFW, free of any analytes of interest that is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures.

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4. Interferences

4.1. Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be necessary.

4.2. Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed sequentially. To reduce carryover, sample syringes must be rinsed with organic-free water following each use. When an unusually concentrated sample is analyzed, it should be followed by the analysis of an instrument blank.

4.3. Contamination may occur by diffusion of volatiles through the septa into the sample during shipment or storage. Analysis of a trip blank and/or a holding blank prepared from organic-free water and carried through the sampling and handling protocol can serve as a check on such contamination.

4.4. All glassware associated with preparation is baked at 50 ±10 °C overnight to further reduce interferences.

4.5. Only high purity reagents and solvents are used to minimize interference.

5. Safety

5.1. The toxicity and carcinogenicity of each reagent used in this method is not precisely defined. However, all compounds and solutions should be treated as health hazards, and exposure of these chemicals to skin and clothing should be minimized to the lowest possible level by whatever means available.

5.2. Skin contact with all chemicals should be minimized by the use of nitrile gloves, safety glasses, and laboratory coats.

5.3. Standard solutions should be handled in the fume hoods to avoid chemical exposure.

5.4. All GC split vents and vacuum pump exhaust are connected to an exhaust vent.

5.5. ARI maintains a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDS) is available to all personnel involved in the chemical analysis. ARI maintains MSDS sheets for all chemicals used in the laboratory. Consult the MSDS whenever you have questions concerning the handling of a potentially dangerous substance. Information also available at www.msdshazcom.com

6. Equipment and Supplies

6.1. Gas chromatograph/mass spectrometer system

6.1.1. Gas chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for purge-and-trap systems and all required accessories, including syringes, analytical columns, autosampler, and gases. The capillary column should be directly coupled to the source of the mass spectrometer.

7.4.2. Internal standards (IS) – The recommended internal standards are chlorobenzene-d5, 1,4-difluorobenzene, 1,4-dichlorobenzene-d4 and pentafluorobenzene. Other compounds that have similar retention times may be used. Prepare an internal standard (IS) working solution so that the IS concentration in a sample will match the mid-point concentration of the instrument calibration curve. Internal standards are added to the sample by an autosampler using an injection loop. The injection loop size varies by instrument. When making the internal standard mix, adjust the concentration to the loop size for each specific instrument.

7.4.3. 4-Bromofluorobenzene (BFB) standard: Prepare a 25 µg/mL solution of BFB in Methanol.

7.4.4. VOA Spiking Standard (VSS) – Used to prepare MS, MSD, LCS and LCSD samples and calibration standards. Certified working standards containing all of the VOA analytes are purchased from a commercial vendor. Purchased standards are combined and/or diluted to prepare the appropriate spiking solution. The spiking solution is also used to prepare calibration standards.

7.4.4.1. The standard is prepared in methanol, with each compound present at 100 µg/mL except Acrolein and the ketones which are at 500 µg/mL.

7.4.4.2. When a targeted analyte is not available in a commercial certified working standard, the appropriate concentration should be determined, and the solution prepared in a water soluble solvent (usually methanol or acetone) at that concentration.

7.4.5. Calibration standards - Calibration standards are prepared from the VOA Spiking Standard at the time of calibration as described in Section 10.2.

7.4.6. Initial Calibration Verification Standard (ICVS) or Second Source Standard (SSS) is a certified working standard containing the same VOA analytes as the VSS but from a different source. The VSS and ICVS (SSS) may be purchased from different vendors or as different lot numbers from the same vendor. This standard is used to verify the concentration of the VOA spiking solution following an initial calibration.

8. Sample Collection, Preservation, Shipment and Storage

8.1. Water samples must be stored at >0 to 6°C and analyzed within 7 days of sampling if not preserved with 1:1 HCl. Preserved aqueous samples must be analyzed within 14 days. Soil samples are to be stored at >0 to 6°C and must be analyzed within 14 days of sampling.

8.2. Unpreserved solid samples for Method 5035 must be analyzed or preserved within two days of sampling.

8.3. A holding blank will be kept in refrigerators 21 and 25 to be analyzed with samples at specified intervals (Section 9.6).

8.4. VOA Chain of Custody

8.4.1. Samples for VOA analysis are delivered to Refrigerator 25 and logged into the VOA lab by sample receiving personnel using the appropriate spaces in Logbook 8009F.

8.4.2. VOA Laboratory analysts acknowledge receipt of the samples using the appropriate spaces in Logbook 8009F.

8.4.3. VOA analysts organize received samples into storage boxes in Refrigerator 21. Following analysis, any un-used sample is returned to its position in the storage boxes. When analyses of samples in a box are complete, the box will be archived in Refrigerator 36 for 30 days prior to disposal. The archival date is recorded in Logbook 8009F.

8.5. Sample dilution: The following techniques are appropriate for diluting samples into the proper concentration range for analysis.

8.5.1. Use screening data, historic project information, sample appearance or other ancillary information to determine appropriate purge or dilution volumes prior to analysis.

8.5.2. The amount a sample may be diluted is determined by the purge volume and final concentration of target analytes.

8.5.2.1. All dilutions >50X should be run using a 5 mL purge volume.

8.5.2.2. Actual sample concentrations should not exceed 60 x 50 = 3000 ppb for a 10 mL purge.

8.5.3. Prepare dilutions in a 45 mL vial taking care to maintain sample integrity by keeping atmospheric exposure of the sample to a minimum

8.5.4. Add a stir bar to the 45 mL vial or leave a small pea bubble in the vial to facilitate mixing while shaking the sample.

8.5.5. Dilutions in excess of 1000X must be prepared using an intermediate Methanol (or OFW) dilution taken directly from the sample vial.

8.5.6. Always perform a final review of multiple analyses from the same sample and note any major deviations in analyte concentrations on the analyst notes Form 8042F.

8.6. Compositing samples prior to GC/MS analysis

8.6.1. The samples must be cooled at >0 to 6°C during this step to minimize volatilization losses. Combine an equal amount of each sample to be composited in a volumetric flask. Invert and shake 3 times and transfer to a sample vial or a 5mL gas tight syringe.

8.6.2. Samples composited in this fashion will be qualified in the analyst notes.

9. Quality Control

9.1. Quality Control procedures and requirements are summarized in Appendix 20.1.

9.1.1. Acceptance criteria for ARI's routine analyses listed in the column title "ARI Acceptance Criteria".

9.1.2. When DoD-QSM acceptance criteria differ they are provided in the "DoD-QSM Acceptance" column

9.2. Some clients and/or projects may have different quality control procedures or criteria. Always read and understand any "special Instructions" supplied by an ARI Project Manager.

9.3. Instrument control:

9.3.1. A Method Detection Limits (MDL) study is performed for each analysis to provide recovery and precision for the method.

9.3.2. The GC-MS must be tuned to the specifications in Appendix 20-4 before samples are analyzed. It is required that a 5-50ng total BFB standard, CCV, LCS, LCSD and a method blank be analyzed with acceptable QC before analyzing samples.

9.3.3. Internal standard (IS) area criteria in the samples must be evaluated for retention time shift and EICP areas.

9.3.3.1. If the EICP area for any IS changes by -50% to +100% from the area of the IS in the mid point of the initial calibration, the samples must be reanalyzed.

9.3.3.2. Retention time shift of internal standards must be ± 0.06 RRT units from the CCV internal standard RT.

9.4. Method performance:

9.4.1. Samples are analyzed in 12 hour run sequences known as QC periods. Each 12 hr QC period begins when a BFB / CCal sample analysis starts and end following the analysis of the last sample injected within 12 hours of the initial sample injection.

9.4.2. One method blank will be performed each 12 hour shift.

9.4.2.1. A method blank is run to demonstrate system cleanliness (no analytes should be detected > ½ reporting limits). Surrogates are to be within the established control limits.

9.4.2.2. Corrective action must be taken when MB contamination is greater than ½ the reporting limits. See section 16.2.

9.4.2.3. Methylene Chloride, Acetone and 2-Butanone are allowed at up to 5 times the reporting limit. Method blanks containing any of these analytes > ½ the RL must be documented in the analyst notes for the analytical run and all associated hits flagged with a "B".

9.4.2.4. Prepare a water method blank by adding 45 mL organic-free water to a pre-cleaned auto-sampler vial.

9.4.2.5. Prepare a soil method blank by adding 5 mL organic-free water to a pre-cleaned auto-sampler vial.

9.4.3. A Laboratory Control Sample must be analyzed before analyzing samples. At a minimum one (LCS) must be run in each 12 hr. QC period. It is recommended an (LCS) duplicate be analyzed also. The control limits for all compounds is ± 20% or ARI's published historical control limits. The maximum allowed relative percent difference (RPD) for an LCS and LCSD) is 20%. Compounds that do not meet these limits must be documented on an Analyst Notes Form (Form 8042F) and included in the data package for review

9.4.3.1. Prepare a water LCS by adding VOA Spiking Standard (VSS) to 45mL organic-free water, to a final concentration equivalent to that of the CVS.

9.4.3.2. Prepare a soil LCS by adding the VSS to 5mL of organic-free water to a final concentration equivalent to that of the CVS.

9.4.4. One set of matrix spikes is analyzed for each 20 samples/matrix/instrument (when requested and adequate sample is available) at a known concentration level within the range curved at 50ng/mL for 5mL and 10ng/mL for 10mL purges and 50ng/g for soils.

9.4.4.1. ARI will not perform MS/MSD analysis on any of the field QC samples delivered as part of a client's QA/QC program (water rinsate samples, field/trip blanks etc.)

9.4.4.2. Dilution of MS/MSD extracts to get either spiked compounds or native analytes on scale is not necessary.

9.4.5. QC limits are provided to bench chemists, managers, and QA review personnel as tools for assessing data quality in real-time at the point of data generation.

9.5. A holding blank will be kept in each volatile refrigerator 21 and 25 and will be analyzed every week. Holding blank data is documented in LIMS.

9.6. Surrogates are added to each sample, blank, and standard, and are used to evaluate the purge and trapping efficiency by measuring recovery. Surrogates are brominated, fluorinated or isotope labeled compounds not expected to be detected in samples.

9.7. Statistical Control- Internal quality control limits for analyte spike and surrogate recoveries and relative percent difference for matrix spike and matrix spike duplicates are statistically generated on an annual basis.

9.7.1. These quality control limits are provided to bench chemists, managers, and QA staff as tools for assessing data quality in real-time at the point of data generation.

9.7.2. Practical considerations relating to their dynamic nature require their presentation in a document separate from this SOP. Current control limits may be found in the ARI LQAP.

9.7.3. All analysts using this SOP must use it in conjunction with Control Limit documentation in order to assess data quality and any possible need for corrective actions.

10. Calibration and Standardization

10.1. Summary of VOA calibration procedure:

Table 02 – Summary of GC-MS Calibration Procedure

Step	Section	Procedure	Manual/Computer
1	10.2	Prepare Calibration Samples	
2	10.3	Verify Instrument Tune	
3	10.4	Analyze Calibration samples	
4	10.5	Target Calculates RRF & %RSD	
5	10.6	Validate analyte response	
6	10.7	Analyze ICV	
7	10.8	Evaluate IS Response	
8	10.9	Verify Retention Times	
9	10.10	Update Analytical Method	

10.2. Prepare Calibration samples:

10.2.1. Using a new unopened vial of VOA spiking solution (VSS) (Section 7.5.4.4), prepare a set of five or more calibration samples containing all target analytes. This is accomplished by spiking separate volumes of VOA free water with increasing volumes of the VOA spike working standard as listed in Table 03. Calibration samples are prepared in 45 mL VOA vials. The volume of the calibration samples must be equal to the volume that will be analyzed to account for purge efficiencies that vary with sample volume. The concentration of analytes in the lowest level sample must less than or equal to the method reporting limit. The concentrations in the calibration standards define the working range of the method. A set of at least 5 calibration standards containing the method analytes is required. One calibration standard should contain each analyte at the concentration of the reporting limit for that compound. The other calibration standards should contain analytes at concentrations that define the range of the method. The remaining concentrations should correspond to the expected range of concentrations found in real samples but should not exceed the working range of the GC/MS system. ARI typically calibrates with between five and eight calibration levels covering the dynamic range of the instrument and meeting the required reporting limit of the project. (note: Acrolein and the ketones are at 5X

concentrations). To prepare a calibration standard, add an appropriate volume of a secondary dilution standard solution to an aliquot of organic-free reagent water. Transfer the contents to a purging device.

10.2.2. Ketones and Acrolein are spiked at 5X the concentration levels of the other analytes due to their poor purge efficiency.

Table 03 – Typical Calibration Solution Concentrations

Standard	Direct Purge 5 mL water 5 g Soil	Direct Purge 10 mL Water
1	1	0.2
2	2	0.5
3	5	1.0
4	10	2.0
5	50	10
6	100	20
7	150	40
8	200	60

10.2.3. Initial calibration standards, continuing calibration standards and surrogates for the soil side of the autosampler are made in a minimum amount of water. The standards are then transferred to 45 mL vials, each containing a magnetic stir bar. Internal standards (IS) and Surrogate standards (SS) are added by the autosampler.

10.2.4. Initial calibration standards, continuing calibration standards and surrogates for the water side of the autosampler are made at volumes for use in 45 mL vials containing magnetic stir bars. The appropriate sample volume of the standard is transferred via the autosampler to a sparger vessel, and purged. The IS/SS solution is added by the autosampler standard syringe.

10.2.5. Surrogates are not curved, they are spiked at the same level for all calibration points.

10.3. Verify that the instrument is properly tuned following the procedure in Appendix 20.4

10.4. Analyze Calibration Samples:

10.4.1. Analyze each calibration sample using the exact conditions and procedure to be used for subsequent sample analyzes.

10.4.2. Print the "CLP Report" using Target™ software and evaluate each target analyte.

10.4.2.1. Verify that the automated routine has properly identified and quantified all peaks.

10.4.2.1.1. Perform any required manual integration as necessary.

10.4.2.2. Verify that the internal standard (IS) is in control.

10.5. Target™ will tabulate relative response factors (RRF), Average RRF, relative standard deviation (RSD) and % RSD using Equations 01 through 04 for each compound relative to its internal standard.

Equation 01:
$RRF = (A_X C_{IS}) / (A_{IS} C_X)$
A_X = Area of the characteristic ion for the compound being measured
A_{IS} = Area of the characteristic ion for the associated Internal Standard
C_{IS} = Concentration of the associated internal standard
C_X = Concentration of the compound being measured

Equation 02:
$\text{Average RRF} = \Sigma RRF_i / n$
where:
RRF_i = the peak response factor for each quantitation peak in the calibration standard
n = the total number of standards (usually 7)

Equation 03:
$\text{RSD} = \text{SD} / (\text{Ave RRF}) = \frac{\sum(RRF_i - \text{Ave RRF})^2) / (n-1))^{1/2}}{\text{Ave RRF}}$
where:
SD = Standard deviation of the response

Equation 04:
$\%RSD = RSD * 100$

10.6. Evaluate Analyte Response.

10.6.1. When the %RSD for a given target is ≤ 20%, the detector response is considered linear and the average RRF may be used to quantify that compound.

10.6.2. When %RSD is >20% for any compound, the analyst may use an alternative method to evaluate the acceptability of the calibration.

10.6.3. The curve must meet minimum RRF requirements noted in Appendix 20.3.

10.6.4. If more than 10% of the compounds included in the initial calibration exceed 20% RSD and do not meet the minimum coefficient of determination of 0.99, for alternate curve fits, the chromatographic system is considered to imprecise for analysis to begin.

10.6.5. For linear and non-linear calibration curves based on a least squares regression (LSR) model the coefficient of determination (COD) r^2 must be > 0.99.

10.6.6. Special care should be taken to monitor the RRF in the lowest calibration standard to ensure adequate sensitivity at the reporting limit. Following examination of the ICal and any corrective action, all compounds not meeting the calibration acceptance criteria must be documented on an Analysts Notes Form (8042F)

10.7. An initial calibration verification (ICV) is performed by analyzing a midpoint calibration standard prepared using the ICVS. ARI will spike the full list of compounds at a mid calibration range concentration. Calibration verification is acceptable when the recovered analytes are within ± 30% (20% for DoD analyses) of the expected concentration. Specific clients or projects may allow or require different calibration acceptance limits. When any analytes are not in the acceptable range corrective action and documentation on an Analyst Notes Form (Form 8042F) is required.

10.8. The internal standard responses and retention times in the continuing calibration standard must be evaluated during or immediately after data acquisition.

10.8.1. If the EICP area for any of the internal standards changes from -50% to +100% from the last mid point concentration of the initial calibration, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. Areas are documented in the daily run log.

10.9. If the retention time for any internal standard changes by more than 10 seconds from the last daily calibration, the chromatographic system must be inspected for malfunctions and corrections must be made, as required.

10.10. Update the analytical method file with updated RT and RRF data.

11. Procedure

11.1. Procedure Summary

Table 04 – Procedure Summary

Step	Process	See Section
1	Project Evaluation	11.2
2	Set or Verify Instrument Operating Parameters	11.3
3	Verify Spiking Solution in the autosampler	11.4

4	Prepare Samples for Analysis	11.5
5	Set up Analytical Run	11.6
6	Initiate sample analysis	11.7
7	Verify Instrument Tune and Calibration	11.8
8	Evaluate Mass Spectra	11.9
9	Evaluate QC Analyses	11.10
10	Export Data to LIMS	11.11

11.2. **Project Evaluation**

11.2.1. Holding times: Review project documentation to determine when sample holding time will expire. If there is any chance the sample will not be analyzed with the required holding time, notify your laboratory supervisor and/or the appropriate project manager.

11.2.2. Special requirements: Non-routine analytical requirement may be required for a specific project or sample. Review all project documentation and make sure you understand any special requirement before proceeding with analysis.

11.2.3. Historic Data may be available for a continuing project or sampling site. Use this data to pre-determine any special sample handling necessary.

11.3. **Set up the GC/MS/Autosampler system as outlined in Tables 06, 07 and 08:**

11.3.1. This should be done prior to the preparation of the sample to avoid loss of volatiles from prepared standards and samples.

Table 06 – Typical Instrument Operating Parameters	
Mass Spectrometer	
Mass scan range	35 – 300 amu
Scan time	1 sec/scan or less
Electron volts	70 volts (nominal)
Gas Chromatogram	
Initial Temperature	43 – 46 °C
Temperature Program	46-240°C at 12°C/min
Final temperature:	240°C, hold 4 min
Source temperature:	According to manufacturer's specs, 150-250°C
Transfer nozzle/line:	180-250°C
Carrier Gas:	Helium at 25-50 mL/min
Purge & Trap	
Purge	8-11 minutes

He flow rate	25 – 50 mL/min
Sample Heater	40 °C
Desorb preheat	250°C
Desorb	1-6 minutes at 250°C
Bake	4-10 minutes at 260°C
Valve temperature	50-110°C
Mount temperature	30-110°C
Line	50-200°C

Table 07 – Typical Autosampler Set-up - Manual Mode

Parameter	Autosampler Options	Recommended Set-up	
		Water	Soil
Flush Volume	5, 10, 15, 20 mL	10	5
Select matrix	Water or Soil	Water	Soil
Standard	Yes or No	Yes	Yes
Sample Volume	5, 10, 15, 20 mL	5 or 10	--
Dilution	0 – 95 %	0	--
Flushes	0 - 10	3	--
Stir Time	0 – 15 min	1	--
Settle Time	0 – 15 min	0	--
Stir Speed	L, M, H	M	--
Desorb Time	0 – 10 min	1	--
Flow Rate	0 – 150 mL/min	40	
Line Heat	25 – 125 °C	--	80
Pre-heat	0 – 10 min	0	0
Water Volume	0 – 10 mL	--	7
Pre-purge	0 – 10 min	0	0
Purge time	0 – 20 min	10	10
Flushes	0 - 10	--	1
Soil Stir	Yes or No	--	Yes
Desorb time	0 – 10 min	--	2
Water Trap Volume	Yes or No	--	0

Table 08 – Typical Autosampler Set-up - Auto Mode

Parameter	Options	Recommended Set-up	
		Water	Soil
Start delay	0 – 99.5 hours	0	0
Cycle time	0 – 199 min	0	24
Auxiliary output	Yes or No	No	No
Last water or soil	Vial number	1 – 30	1 - 30
Blank Last	Yes or No	No	No
Flush Volume	5, 10, 15, 20 mL	5 or 10	5
Select Matrix		Water	Soil
Standard	Yes or No	Yes	Yes
Sample Volume	5, 10, 15, 20 mL	5 or 10	--
Dilution	0 – 95 %	0	--
Flushes	0 - 10	2	--
Stir Time	0 – 15 min	1	--
Settle Time	0 – 15 min	0	--
Stir Speed	L, M, H	M	--
Desorb Time	0 – 10 min	1	--
Flow Rate	0 – 150 mL/min	40	40
Line Heat	25 – 125 °C	--	80
Pre-heat	0 – 10 min	--	0
Water Volume	0 – 10 mL	--	7
Pre-purge	0 – 10 min	0	0
Purge time	0 – 20 min	10	10
Flushes	0 - 10	--	1
Soil Stir	Yes or No	--	Yes
Desorb time	0 – 10 min	--	2
Water Trap Volume	Yes or No	--	No

11.4. **Verify Spiking Solutions**

11.4.1. Verify the IS / SS standard volume in the autosampler reservoir. Add standard as necessary.

11.5. **Prepare Samples for Analysis:** Samples must be properly prepared for analysis using the processes outlined in Table 05.

Table 05 – Sample Preparation Procedures

Sample Matrix	Method	Technique	Details in Section:
All	Sample Screening	Sample Dilution	11.5.2
Water	5030B	Direct P&T	11.5.4
Soil	5035	Direct P & T	11.5.5
Solid	5035	Methanol Extraction	11.5.5
Soil		From Total Solids Jar	11.5.3.2
Waste	3585	Waste Dilution	11.5.5.4

11.5.1. **Prepare the Instrument:** The Purge and Trap GC-MS instrument must be readied before samples are prepared for analyses to allow minimum time lapse between preparation and analysis.

11.5.2. **Sample screening:**

11.5.2.1. Any sample with characteristics (color, odor, client information etc.) indicating it may contain high levels should be screened prior to analysis. Screening may also help prevent un-necessary contamination of the purge-and-trap system. Samples are screened by analyzing them at dilution.

11.5.2.1.1. Aqueous samples: dilute the samples with an appropriate volume of OFW and analyze as a normal sample.

11.5.2.1.2. Extract solid samples with methanol and dilute a small aliquot of the methanol into 45 mL OFW for analysis.

11.5.2.1.3. A portable PID may be used to asses the sample.

11.5.3. All client samples, QA samples and standards must be spiked with surrogate (SS) and internal standards (IS) prior to analysis. The SS and IS are normally added to the sample by the auto-sampler. When samples are analyzed manually, the analyst must spike each sample individually.

11.5.4. **Aqueous Sample Preparation:**

11.5.4.1. Screen aqueous samples when historic data or their appearance indicated that they may contain high concentrations (> 2mg/L) of volatile compounds. When the screening indicates a high concentration of volatiles dilute the sample with OFW.

11.5.4.2. Aqueous samples are normally received by ARI in 45 mL VOA vials. The VOA vials are placed directly on the auto-sampler for analysis. The auto-sampler will spike surrogate and internal standard into the sample prior to the purge process.

11.5.4.3. Manual Sample Preparation: Remove the plunger from a 5mL syringe and attach a closed syringe valve. If lower detection limits are required, use a 25mL syringe. Open the sample or standard bottle, which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 or 10 mL. Transfer remaining sample to a 45mL VOA vial with a Teflon™ sealed cap or fill a second syringe at this time to maintain sample integrity.

11.5.4.4. For matrix spike, matrix spike duplicate, and LCS analyses, add the appropriate amount of spiking solution to the 45mL vial containing the sample to be purged to a known concentration level within the range curved.

11.5.5. **Solid Samples (Soil & Sediment) Preparation:**

11.5.5.1. Screen solid samples when historic data or their appearance indicated that they may contain high concentrations (> 2mg/L) of volatile compounds. When the screening indicates a high concentration of volatiles use less sample for a direct purge analysis or use less Methanol extract in the analysis.

11.5.5.2. Solids samples are normally reported on a dry weight basis. Always perform a dry weight determination (Appendix 20.8) unless the project plan requires data reported on an "as received" basis.

11.5.5.3. Low concentration direct purge & trap

11.5.5.3.1. This is designed for samples containing individual purgeable compounds of < 1 mg/kg. The low-concentration method is based on purging a heated (40°C) sediment/soil sample mixed with OFW containing the surrogate and internal standards. Analyze all blanks and standards under the same conditions as the samples.

11.5.5.3.2. Use a 5g sample if the expected concentration is <0.2 mg/kg or a 0.5g-5g sample for expected concentrations between 0.2 and 2mg/kg.

11.5.5.3.3. The sample consists of the entire contents of the sample container. Do not discard any supernatant liquids. If sample has free liquid, mix the unopened container 1 min using a Vortex mixer or mix the contents with a small metal spatula. When no free liquid is present remove the top layer to expose uncompromised sample. Weigh the sample into a tared purge device and record the actual weight to the nearest 0.1g. Samples collected following EPA Method 5035 will be preweighted.

11.5.5.3.4. Heat and purge the sample. Be sure the trap is cool (<35°C)

11.5.5.3.5. If saturated peaks occurred or would occur if a 0.5g sample were analyzed, the high concentration method (Medium Level soil method) must be followed.

11.5.5.4. Methanol Extraction for medium level soil or waste samples. This method is based on extracting the sediment/soil with methanol. A waste sample is either extracted or diluted, depending on its solubility in methanol.

11.5.5.4.1. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Using a top loading balance weigh 5g (wet weight) of sample into a tared 20 mL vial. Record the actual weight to 0.1g. Determine the percent dry weight of the sample as described in Appendix 20.4. Measure 5mL of Methanol into the vial. Stir the sample Methanol mixture for 2 to 3 minutes using a vortex mixer.

11.5.5.4.2. Pipet a portion of the extract to a clean amber Teflon sealed vial for storage (limiting headspace). The remainder may be disposed. Store the extracts at >0 to 6°C in refrigerator 25, prior to analysis.

11.5.5.4.3. After determining the required dilution, add the appropriate amount of extract to the autosampler vial (100µL to 10µL / 5mL). If the extract amount to be added is less than 10µL, or a secondary dilution is needed, surrogates will be diluted to a level near or below the calibration range; additional surrogate must be added by the autosampler standard syringe.

11.5.5.4.4. For a matrix spike in the medium level samples, add 25µL of matrix spike solution and an appropriate aliquot of this extract to 5mL of organic-free water for purging. Purge and start analysis of the sample.

11.6. **Set Up Analytical Run**

11.6.1. Samples are analyzed in 12 hour run sequences known as QC periods. Each 12 hr QC period begins when a BFB / CCal sample analysis starts and end following the analysis of the last sample injected within 12 hours of the initial sample injection.

11.6.2. Setup an analytical run by placing 45 mL sample vials containing either standards or client samples in the autosampler tray in the order listed in Table 09.

Table 09 – Example Analytical Run Sequence

Sample Sequence	Sample Type
1*	BFB Tuning sample
2*	Continuing Daily Calibration
3	LCS
4	LCSD
5	Method Blank
6 through 50	Prepared Client Samples
* These may be combined	

11.6.3. The autosamplers may run in manual or automatic mode.

11.6.4. Enter the sample identifications into the Chemstation software

11.7. **Initiate Sample Analysis**

11.7.1. Proceed with the analysis. Analyze all blanks on the same instrument as that used for the samples. The standards and blanks should also contain 100µL of the purge-and-trap grade MeOH to simulate the sample conditions when analyzing extracted solid samples.

11.7.2. The instrument system will transfer sample to the purge and trap sampling device and perform the GC-MS analysis automatically.

11.7.3. The analyst is responsible for assuring that the automated peak identifications and integrations are acceptable.

11.8. **Verify Instrument Tune and Calibration**

11.8.1. The 12 hr QC period starts with the BFB injection time and ends with the injection time of the last run inside the 12 hours. BFB and CVS may be combined as long as both criteria can be met.

11.8.2. Review chromatographic data to assure all peaks are identified and integrated correctly following the procedures in Appendix 20.2.

11.8.3. Verify that the response of all internal standards is -50% to +100% of the compound's response in the most recent initial calibration mid point standard.

11.8.4. The GC/MS system must be hardware tuned to meet the ion abundance criteria in Appendix 20.4 for analysis of ≤ 50 ng BFB. Analysis must not begin until all criteria have been met. Compliance with the criteria must be demonstrated every 12 hours at a minimum.

11.8.4.1. Evaluate the BFB in the following manner.

11.8.4.1.1. Acquire and average the entire peak (all scans) or three scans (the peak apex scan and the scans immediately preceding and following the apex).

11.8.4.1.2. Subtract background, this is required and must be accomplished using a single scan no more than 20 scans prior to the elution of BFB. **Do not subtract part of the BFB peak.**

11.8.5. Daily GC/MS calibration and calibration check verification

11.8.5.1. A calibration check standard at mid-concentration containing each compound of interest, including all required surrogates, must be performed once every 12 hours prior to sample analysis. Compare the response factor data of the standard each 12 hour shift that samples are to be analyzed against the average response factor from the initial calibration for a specific instrument.

11.8.5.2. Determine the percent drift (%D) for all analytes in the daily continuing calibration.

11.8.5.3. The calibration for all compounds with a %D ≤ 20 is acceptable.

11.8.5.3.1. Method 8260 allows up to 20% of the target analytes to be greater than 20%. If more than 20% of the analytes have %D > 20% corrective action is required prior to analysis.

11.8.5.3.2. Compounds with >20%D may be reported when it can be demonstrated that there is adequate sensitivity to detect the compound if it were present. Such compounds must be documented on an Analysts Notes Form (Form 8042F) and the data Q-flagged for any reported value.

11.9. **Evaluate Mass Spectra**

11.9.1. Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum. If not, the compound may be flagged with "M" if the analyst feels the identification is real (this favors false positives).

11.9.2. The relative intensities of the major ions should generally agree with the reference spectra.

11.9.3. Molecular ions present in the reference spectrum should be present in the sample spectrum.

11.9.4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

11.9.5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

11.9.6. An analyte is identified by comparison of the sample mass spectrum with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the initial calibration. These standard reference spectra may be obtained through analysis of the calibration standards. The characteristic ions are defined as the three ions of greatest intensity, or any ions over 30% intensity relative to the base ion, if less than three such ions occur in the reference spectrum. Two criteria must be satisfied to verify identification: (1) elution of sample component at or near the same GC relative retention time (RRT) as the standard component; and (2) correspondence of the sample component mass spectrum and the standard component mass spectrum.

11.9.7. The intensities of the characteristic ions must maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing

ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

11.9.8. The sample component RRT must compare within ±0.06 units of the RRT of the standard component. For reference, the standard must be run within the same 12 hour QC period as the sample. If co-elution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted, ion-current profiles for ions unique to the component of interest.

11.9.9. All ions present in the standard mass spectra at a relative intensity greater than 10% (the most abundant ion in the spectrum is equal to 100% intensity) should be present in the sample spectrum.

11.9.10. The relative intensities of ions specified in Appendix 20.3 must agree within plus or minus 30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample abundance must be between 20% and 80%.). If not, the compound may be flagged with an "m" if the analyst determines that the identification is valid (favors false positive).

11.9.11. Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs and reported as the sum of both compounds.

11.9.12. Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes co-elute (i.e., only one chromatographic peak is apparent), the identification criteria can be met, but each analyte spectrum will contain extraneous ions contributed by the co-eluting compound.

11.9.13. Secondary ion quantitation is allowed only if there are sample matrix interferences with the primary ion.

11.9.14. All dilution efforts should try to keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve. To determine the dilution factor, compare a minor ion in the saturated analyte against the daily standard.

11.10. **Evaluate the QA samples as outlined in Section 12**

11.10.1. Daily GC/MS calibration verification, LCS criteria, and the MB must demonstrate the system is free of interferences, before analyzing samples.

11.10.2. If the analysis shows the sample to have a concentration of analytes that exceed their highest standard, the sample must be rerun at a dilution. Analyst considerations for chromatographic "system overload" are analytes that are above the linear range, saturation of the mass spectrometer, chromatography overload, or analyte/interference carryover. Professional judgment must be used by the analyst as to the modification of the analytical sequence. If system contamination is present, an OFW blank must be analyzed. If the blank analysis is not free of analytes/interferences, the system should undergo maintenance, such as baking the trap or purging with methanol to decontaminate the system. If a blank is not run, as with auto-samplers, the following sample must be checked for carryover and rerun if it contains the same compounds which showed saturation. This must continue until no analyte is detected > ½ the RL.

11.11. **Export data to LIMS**

12. Data Analysis and Calculations

12.1. Quantitative analysis

12.1.1. It is the operator's responsibility to verify all compound identifications performed by the GC-MS data system. Use retention time, spectral data, data system calculated fit, and the operator's expertise to determine whether analytes found by the system are real.

12.1.2. When a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. Quantitation will take place using the internal standard technique. The internal standard used shall be the one nearest the retention time of that of a given analyte.

12.1.3. If secondary ion quantitation is necessary due to interference, then a short quantitation report list is generated. This quantitation contains the integrated areas of the affected compounds, based on the secondary ion(s) for that compound, and of the relevant internal standards. Identical reports must be generated for the sample with interference and for the relevant continuing calibration. The report for the continuing calibration is used to generate a relative response factor for the affected compound based on its secondary ion. This relative response factor is then used in the calculations for that compound in the affected sample. The short quantitation report may be hand calculated by the analyst as long as it is signed and dated by the analyst.

12.1.4. The concentration of each identified analyte in the sample is calculated as follows:

12.1.4.1. Aqueous samples

Concentration (µg/L) =	$\frac{(Ax)(Is)}{(Ais)(RF)(VO)}$
where:	
Ax =	Area of characteristic ion for compound being measured.
Is =	Amount of internal standard injected (ng)
Ais =	Area of characteristic ion for the internal standard.
RF =	Response factor for compound being measured.
VO =	Volume of water purged (mL), taking into consideration any dilutions made.

12.1.5. Sediment /Soil, Sludge, and Waste:

12.1.5.1. High-concentration:

Concentration (µg/Kg) =	$\frac{(Ax)(Is)(Vt)}{(Ais)(RF)(Vi)(Ws)}$
where:	
Ax =	Area of characteristic ion for compound being measured.
Is =	Amount of internal standard injected (ng)
Vt =	Volume of total extract (L) (use 5,000µL or a factor of this when dilutions are made).
Ais =	Area of characteristic ion for the internal standard.
RF =	Response factor for compound being measured.
Vi =	Volume of extract added (L) for purging
Ws =	Weight of sample extracted or purged (g). The wet weight or dry weight may be used depending upon the scientific applications of the data.

12.1.6. Low-concentration

Concentration (µg/Kg) =	$\frac{(Ax)(Is)}{(Ais)(RF)(Ws)}$
where:	
Ax =	Area of characteristic ion for compound being measured.
Is =	Amount of internal standard injected (ng)
Ais =	Area of characteristic ion for the internal standard.
RF =	Response factor for compound being measured.
Ws =	Weight of sample extracted or purged (g). The wet weight or dry

weight may be used depending upon the scientific applications of the data.

12.1.7. Sediment/soil samples are generally reported on a dry weight basis (sludge and wastes are also reported on a dry weight basis). In either instance, the percent dry weight of the sample should be reported along with the data.

12.1.8. Background organic material in sample extracts, usually due to hydrocarbons typically manifests itself in the form of a broad peak or peaks in the chromatogram. When mandated by project-specific requirements, it is necessary to provide both a tentative identification and an approximate concentration for such peaks, utilizing the following procedures.

12.1.9. A tentative spectral identification for such a peak is established by comparison of multiple spectra from the peak with those in the NIST library. A minimum of three un-enhanced scans, one each from near the beginning, middle and end of the peak shall be used. An EICP containing the three most abundant ions common to each of the three spectra shall be plotted for the region of the chromatogram encompassing the peak.

12.2. Analysis records

12.2.1. Each analytical run will be recorded in the instrument run log, including the vial ID and pH of each sample for aqueous samples. This log book serves as a routine maintenance log and the chain-of-custody for analyzed samples. Any change of operator must be included in the notations.

12.2.1.1. Fill in the date, your name, and tune file in the run log. For each standard and analytical run, fill out the file name, and if space is available, client sample number and sample amount.

12.2.2. Soil extractions and 5035 analyses are noted on the Volatile Organics Extraction Bench Sheet 8043F, which is included with the analysis data.

12.2.3. Total solids are recorded on the Total Solids bench Sheet 5050F, and included with the analysis data.

13. Method Performance

13.1. Method performance is verified daily by comparison with historical data and through annual MDL studies. This data is available on the ARI intranet.

13.2. The QA department measures method performance using a combination of annual method detection limit (MDL) studies, performance evaluation samples, and the monitoring of surrogate and spike recoveries.

13.2.1. Reporting limits for all analytes quantitated using this SOP are set using the low point of the initial calibration curve and validated by method detection limit studies.

13.2.2. MDL studies are performed each year for each analyte.

13.2.3. MDL and reporting limit (RL) values may be found for each analyte in the ARI intranet.

13.3. Laboratory precision and bias measurements are performed by monitoring surrogate and spike recoveries in client and quality control samples.

13.3.1. Control limits are calculated by monitoring these recoveries. These control limits are disseminated to the bench chemists and LIMS administrator for use in monitoring method performance in real time. As these limits are updated regularly, their dynamic nature prevents their inclusion in this SOP. However, they may be found in the ARI intranet.

14. Pollution Prevention

14.1. A hazardous waste satellite accumulation station is provided for disposal of all solvent or potentially hazardous samples.

14.2. All syringe rinsing must be performed over charcoal to minimize the exposure of the environment to solvent.

14.3. All GC split vents will be connected to an exhaust vent.

14.4. All MS vacuum pumps will have a charcoal exhaust filter.

14.5. Autosampler waste is neutralized *in situ* using limestone chips.

14.6. All activated charcoal containers must be covered when not in use.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. Requirements relating to initial and continuing calibration are detailed in Section 10 of this document.

15.2. Method Blanks- The method blank must contain less than 1/2 the reporting limit of the targeted analytes or corrective action is required.

15.3. Internal Standards- All samples' internal standard EICP areas following the continuing calibration standard must meet the technical acceptance criteria listed in Section 9.7 .

15.4. Surrogate Recoveries

15.4.1. All method blanks, laboratory control samples, matrix spikes, matrix spike duplicates, duplicates or other samples must have acceptable surrogate recoveries. Surrogate recoveries are considered unacceptable when:

15.4.1.1. Any surrogate has a recovery that is outside ARI or project specific control limits.

15.4.2. These requirements do not apply to subsequent analysis of samples where a prior analysis of the sample shows unacceptable surrogate recovery. This may demonstrate a matrix effect.

15.4.3. When mandated by contract-specific requirements, corrective actions must be performed in response to failure to meet surrogate acceptance criteria, even when we meet in house limits.

15.4.4. Surrogate recovery acceptance windows are ideally determined statistically from method and matrix specific laboratory data updated on a periodic basis. Certain methods or clients may specify project specific surrogate recovery acceptance windows.

15.4.5. Surrogate acceptance criteria are both matrix and concentration level specific (e.g. low level vs. medium level soils). When analyzing matrices or concentration levels for which no acceptance criteria are available, the closest approximation of available acceptance criteria may be provided as estimates for advisory purposes only.

15.5. Laboratory Control Samples (LCS)

15.5.1. The LCS recovery values should fall within the specified recovery acceptance limits. If an LCSD is performed then relative percent difference (RPD) acceptance limits may also apply, if available.

15.5.1.1. Marginal Exceedances of LCS compounds are addressed in the LQAP in the Control Limit Marginal Exceedance Policy and must be documented in the analyst notes.

15.5.2. LCS recovery acceptance windows are ideally determined statistically from method and matrix-specific laboratory data updated on a periodic basis. Project or method specific limits may supersede laboratory acceptance criteria.

15.6. Matrix Spike/Matrix Spike Duplicates (MS/MSD)

15.6.1. Matrix Spike/Matrix Spike Duplicate recovery values should fall within the specified recovery acceptance limits. If a MSD is performed then relative percent difference (RPD) acceptance limits may also apply, if available.

15.6.2. MS/MSD recovery and RPD acceptance is advisory and data should not necessarily be rejected based upon MS/MSD recovery. MS/MSD recovery should be compared to LCS/LCSD recovery to determine if recovery trends are present. Certain projects or clients may require project specific MS/MSD recovery and RPD acceptance windows.

15.7. Holding Times

15.7.1. Samples should be run within holding times (seven days for unpreserved water samples and fourteen days for solid samples, MeOH extracts and preserved water samples).

15.7.2. In the event that re-analysis due to an out of control event requires that samples be re-analyzed after their holding time has elapsed the analyst should analyze and report both data sets, whenever practical, distinguishing between the initial analysis and re-analysis on all deliverables. This will document that the samples were originally analyzed within holding

times and may allow for comparisons that will determine whether any of the more volatile analytes were lost in the interval between analyses.

16. Corrective Actions for Out of Control Events

16.1. The analyst must provide a narrative of the volatile analysis in the Analyst Notes. Corrective actions, quality control, and other sample-specific information must be included.

16.2. Method Blanks- Corrective action for a method blank which fails acceptance criteria in Appendix 20,1 may involve reanalysis of all associated samples and/or "B" flagging of the associated sample data. Each occurrence will be evaluated on an individual basis upon consultation with the Project Manager, the client, the Laboratory Supervisor, and the Laboratory Manager.

16.3. Mass Spectrometer Tuning

16.3.1. When the MS does not produce an acceptable mass spectrum when injected with 5 to 50 µg/mL of BFB, re-inject the BFB. If the spectrum again fails to meet the criteria found in Appendix 20.4, the MS must be re-tuned.

16.3.2. If the re-tuned mass spectrum still fails to meet the criteria found in Appendix 20.4, the GC-MS may require maintenance. Maintenance may include replacing the filaments, cleaning the MS source, cleaning the MS lenses, or replacing the electron multiplier.

16.4. Internal Standards

16.4.1. If the internal standards fail to meet their acceptance criteria corrective action must be taken.

16.4.1.1. Corrective action may not be required when all target analytes associated with the failed IS are not detected, if the IS fails high.

16.4.1.2. Check the calculations and correct as necessary.

16.4.1.3. Check the IS compound spiking solutions. If the internal standard compound spiking solution was improperly prepared, concentrated, or degraded, re-prepare solutions and reanalyze the samples.

16.4.1.4. Verify the instrument operation. If the instrument malfunctioned, correct the instrument problem and reanalyze the sample. This correction will mostly likely involve instrument maintenance similar to the maintenance described in Section 16.3.2. If the instrument malfunction affected the calibration, recalibrate the instrument before reanalyzing the sample extract.

16.4.2. If the above actions do not correct the problem, then the problem may be due to a sample "matrix effect". To determine if there is a matrix effect, take the following corrective action steps.

16.4.2.1. Reanalyze the sample. EXCEPTION: if the internal standard compounds responses and/or retention times in a sample used for a matrix spike and/or matrix spike duplicate were outside the acceptance criteria, then it should be reanalyzed only if the internal standard responses and/or retention times were within the acceptance criteria for the associated matrix spike (MS only) or for both the matrix spike and matrix spike duplicate analysis (MS/MSD). These corrective actions are also to be applied in the case of un-spiked duplicates of a given sample which is outside acceptance criteria.

16.4.2.2. If the corrective actions listed above do not prove matrix effect, then the problem with the initial analysis is deemed to be within the laboratory's control. Submit either both sets of data or only the data from the reanalysis with the internal standard responses and retention times within acceptance limits.

16.4.2.3. If the corrective actions listed above prove matrix effect, then submit data from both analyses, distinguishing between the initial analysis and reanalysis on all deliverables.

16.4.2.4. If internal standard acceptance criteria are not met in a sample judged by the GC/MS analyst to have excessive background (e.g. hydrocarbons, high concentrations of non-target analytes and solvents) which may damage the analytical system, the sample may be diluted prior to the reanalysis performed to prove matrix effect.

16.4.2.5. If several samples from a sample delivery group or collected from the same location are similar based on the total ion chromatogram, it may be appropriate to reanalyze a subset (one or more) of the samples to prove matrix interference. Consult the Laboratory Manager or Project Manager to determine if this option is viable.

16.5. Surrogates

16.5.1. When a surrogate compound fails to meet recovery acceptance criteria, reanalyze the sample.

16.5.1.1. It is not necessary to re-analyze a sample when all compounds associated with a high surrogates are not detected.

16.5.2. When the surrogate recoveries are not acceptable in the re-analysis:

16.5.2.1. Verify the calculations and correct as necessary.

16.5.2.2. Examine the sample preparation logs, if preparation logs indicate that the incorrect amount of surrogate compound spiking solution was added either re-calculate surrogate recoveries based on the actual amount of surrogate compound spiking solution added, or re-extract/reanalyze the sample adding the correct amount of surrogate spiking solution.

16.5.2.3. Check the surrogate compound spiking and calibration solutions and If the surrogate compound spiking solution and/or surrogate calibration solution was improperly prepared, concentrated, or degraded, re-prepare solutions and re analyze samples.

16.5.3. If the surrogate compound recoveries meet acceptance criteria in the reanalyzed sample, then the problem with the initial analysis is deemed to be within the laboratory's control. Therefore, submit data only from the reanalysis if the re-analysis was performed within holding time, otherwise report both sets of data.

16.5.4. If the above actions do not correct the problem, then the problem may be due to a sample matrix effect.

16.5.4.1. If the surrogate compound recoveries fail to meet the acceptance criteria in the reanalyzed sample, then submit data from both analyses, distinguishing between the initial analysis and the reanalysis on all deliverables.

16.6. Laboratory Control Samples

16.6.1. When LCS compounds fail to meet their recovery, marginal exceedance or RPD (if applicable) acceptance criteria reanalyze the LCS.

16.6.2. If the LCS recoveries do not meet their acceptance criteria after reanalysis, corrective action is required.

16.6.2.1. Check calculations and correct as necessary

16.6.2.2. Check the sample preparation logs and if the sample preparation logs indicate that the incorrect amount of LCS compound spiking solution was added either recalculate LCS recoveries based on the actual amount of LCS compound spiking solution added or reanalyze the samples, adding the correct amount of LCS spiking solution.

16.6.2.3. Check the LCS compound spiking and calibration solutions and if the LCS spiking or LCS calibration solutions were improperly prepared, concentrated, or degraded, re-prepare solutions and reanalyze samples.

16.6.2.4. Verify proper instrument operation. If the analytical instrument malfunctioned, correct the instrument problem and reanalyze the sample extract. This correction will probably involve maintenance similar to the maintenance discussed in Section. If the instrument malfunction affected the calibration, recalibrate the instrument before reanalyzing the sample extract.

16.6.3. If the LCS compounds still fail to meet their acceptance criteria, reanalyze the LCS and all associated samples and QC samples if deemed appropriate (i.e. after consideration of all batch QC data) or mandated by contract-specific requirements. Any decision to forgo recalibration/reanalysis based on failure to meet LCS acceptance criteria will require approval of the Project Manager and the Lab Manager, at a minimum.

16.7. Sample dilution is required when any analyte exceeds the working range of its calibration. All target analytes must be quantified within the working range of their calibration.

16.7.1. All dilutions should keep the response of the major constituents in the upper half of the linear range of the curve.

16.7.2. When ions from a target compound saturate the detector the analyst must:

16.7.2.1. Flag all affected analytes with an S flag.

16.7.2.2. Analyze an OFW instrument blank until the system has been decontaminated.

16.7.2.3. When an instrument or solvent blank contains interferences, the chromatographic system must be decontaminated before an analytical sequence is resumed.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. See Section 16.3 for guidance on dealing with out-of-control tuning events.

17.2. See Section 16.4 for guidance on dealing with internal standard out-of-control events.

17.3. See Section 16.5 for guidance on dealing with surrogate out-of-control events.

17.4. See Section 16.2 for guidance on dealing with method blank related out-of-control events.

17.5. See Section 16.6 for guidance on dealing with LCS related out-of-control events.

17.6. See Section 16.7 for guidance on dealing over range samples.

18. Waste Management

18.1. All sample vials containing Methanol must be disposed of by placing them in the blue hazardous waste drum in the lab set aside for this purpose. No vials may be thrown in the trash or receptacles not expressly designated for this purpose.

18.2. All standard vials must be disposed of by placing them in the blue hazardous wasted drum in the lab set aside for this purpose.

18.3. All solvents must be disposed of by pouring them out over charcoal. No solvent may be poured down the drain or disposed of in any other non-hygienic manner.

18.4. All spent charcoal must be disposed of by placing it in the charcoal disposal bin located in the extractions lab.

18.5. Autosampler waste is neutralized *in situ* using limestone chips.

18.6. All acid preserved vials must be neutralized and logged in the Neutralization Log.

19. Method References

19.1. "Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Method 8260C Revision 3, August 2006.

19.2. USEPA Contract Laboratory Program Statement of Work for Organics Analysis, Multi-Media, Multi-Concentration Revision OLM03.1, August, 1994.

19.3. "Determinative Chromatographic Separations": Method 8000C, Test Methods for Evaluating Solid Waste (SW-846), Revision 3, March, 2003.
19.4. Department of Defense (DoD) Quality Systems Manual – Version 3 Final 5 June 2003
19.5. "Sample Preparation for Volatile Organic Compounds" EPA Method 5000, Revision 0, December 1996

20. Appendices

20.1. Appendix 20.1: Quality Control Requirements
20.2. Appendix 20.2: Manual Integration
20.3. Appendix 20.3: 8260C target analyte list
20.4. Appendix 20.4: BFB ion abundance criteria
20.5. Appendix 20.5: Chromatogram of Typical VOA calibration standard
20.6. Appendix 20.6: Example page from GC/MS VOA organics logbook
20.7. Appendix 20.7: Sample Screening
20.8. Appendix 20.8: Dry weight determination
20.9. Appendix 20.9: Tentatively Identified Compounds
20.10. Appendix 20.10: Pea Bubble Chart

Appendix 20.1 - Method 8260C Quality Control Requirements

Appendix 20.1 - Method 8260C Quality Control Requirements

QC Check	Minimum Frequency	Acceptance Criteria	DoD Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see ARI SOP 1017S)	QC acceptance criteria published by DoD, if available; otherwise method specified criteria.	Same	1) Recalculate results 2) Locate and correct the source of the problem and repeat the test for all parameters of interest.	Not applicable (NA)	This is a demonstration of ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample) as described in ARI SOP 1017S. An analyst must completed a successful demonstration of capability before analyzing client samples
Method detection limit (MDL) study	At initial set-up and subsequently once per 12 month period.	See 40 CFR 136B. MDL verification checks must produce a signal at least 3 times the instrument's noise level.		Run MDL verification check at higher level and set MDL higher or reconduct MDL study	NA	Samples cannot be analyzed without a valid MDL.
Tuning	Prior to calibration and every 12 hours during sample analysis	Refer to method for specific ion criteria. Option A or B see appendix 20.4	Method specific tuning criteria from 8260C option B must be used.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate	Problem must be corrected. No samples may be accepted without a valid tune.
Evaluation of relative retention times (RRT)	With each sample	RRT of each target analyte in each calibration standard within ± 0.06 RRT units of the daily CCal	DoD requires re-analysis of the ICal	Correct problem, then rerun CCal.	Flagging criteria are not appropriate.	
Minimum five-point Initial calibration for all analytes (ICAL)	Initial calibration prior to sample analysis	Option 1: RSD for each analyte ≤ 20% Option 2: linear least squares regression $r^2 > 0.99$ Option 3: non-linear regression - coefficient of determination (COD) $r' \geq 0.99$ (Max 10% of target analytes may fail) (6 points shall be used forsecond order)	Same	Correct problem then repeat initial calibration.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification	Once after each initial calibration	Value of second source for all analytes within ± 30% of expected value (initial source)	Value of second source for all analytes within ± 20% of expected value (initial source)	Correct problem, verify second source standard. Rerun verification. If that fails, correct problem and repeat ICal	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.

Appendix 20.1 - Method 8260C Quality Control Requirements

QC Check	Minimum Frequency	Acceptance Criteria	DoD Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention time established for all analytes and surrogates	Once per ICAL and at the beginning of the analytical shift	Position shall be set using the midpoint standard of the calibration curve or the value in the CCV run at the beginning of the analytical shift.	Same	NA	NA	
Calibration verification (CV)	Daily, before sample analysis, and every 12 hours of analysis time	%Difference/Drift for all target analyes: ≤ 20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration.) Up to 20% of analytes may exceed ≤ 20%D	1. Average RF for SPCCs: VOCs ≥ 0.30 for chlorbenzene and 1,1,2,2-tetrachlorethane; ≥ 0.1for chloromethane, bromoform, and 1,1-dichloroethane. 2. %D ≤ 20%D for all analytes.	Correct problem, then rerun CV. If that fails, repeat initial calibration.	DoD: Apply Q-flag if no sample material remains and analyte exceeds criteria. ARI: Apply Q-flag to any analytes that exceed ≤ 20%D	.
Internal standards verification	In all field samples and standards	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL EICP area within - 50% to + 100% of ICAL midpoint standard	Same	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.
Method blank	One per preparatory batch	No analytes detected > ½ RL. For common laboratory contaminants, no analytes detected ≥ 5 X the RL.	No analytes detected > ½ RL. For common laboratory contaminants, no analytes detected > the RL.	Correct problem, if required, reprep then reanalyze method blank and all samples processed with the contaminated blank.	Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch	
Laboratory control sample (LCS) containing all reported analytes & surrogates	One LCS per preparatory batch	QC acceptance criteria specified published in ARI's LQAP.	Same unless QC acceptance criteria specified by DoD.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated batch, if sufficient sample material is available	If corrective action fails apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch	
Matrix spike (MS)	One MS/MSD per preparatory batch per matrix when sufficient sample is available	For matrix evaluation, use QC acceptance criteria specified by for LCS.	Same unless QC acceptance criteria specified by DoD.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are out of control, eval- uated data to determine the source of difference and to determine if there is a matrix effect or analytical error.

Appendix 20.1 - Method 8260C Quality Control Requirements

QC Check	Minimum Frequency	Acceptance Criteria	DoD Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate spike	All field and QC samples	QC acceptance criteria specified by DoD, if available; otherwise method-specified criteria or laboratory's own in-house criteria	Same	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associa- ted preparatory batch, if sufficient sample is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	For the specific analyte(s) in all field samples collected from the same site matrix as the parent, apply J-flag if acceptance criteria are not met. For QC samples, apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Results report- ed between LOD & LOQ	NA	NA	NA	NA	Apply J-flag to all results between LOD and LOQ.	

Appendix 20.2

Manual Integration

Manual Adjustment: - Modern chromatographic instruments include computer software to identify a detector response as a chromatographic peak, characterize that peak and determine the relative height or area of the signal. The software utilizes parameters (threshold, slope, etc.) that are adjusted by the instrument operator to optimize the results. A single set of operator controlled settings that determine peak characteristics for an entire data file (or sets of data files) is defined as an "automated procedure." An automated procedure often characterizes chromatographic peaks incorrectly. ARI requires that trained analysts identify and resolve these errors using an alternative automated procedure or a "manual adjustment" of the data. Manual adjustment is defined as the process used by an analyst to adjust an individual peak or a subset of data in a chromatographic file.

The settings for the routine automated procedure normally used to process chromatographic data for this SOP are described below for both the total (or reconstructed) ion chromatogram (RIC) used for the quantitation of TIC's and for the extracted ion current profile (EICP) used for the quantitation of target compound analytes and surrogates.

Parameter	RIC	EICP
Peak Detection - Start	0.200	0.200
Peak Detection - End	0.000	0.000
Bunch	1	1
Peak Smoothing	on	off
Area Cutoff	0 %	5 %
Maximum Peaks to Detect	200	100
Baseline Reset	3 - 5	5
Set Valley	100	100

Trained analysts may subsitute one automated procedure for another in order to optimize peak characteristics. The use of an alternate automated procedure must be permanently documented using either a software generated log file or analyst notes.

Manual adjustment of chromatographic peak characteristics will be used to correct the results of an automated procedure that, in the trained analyst's opinion, are clearly incorrect and will result in erroneous peak identification, integration or quantification.

Manual adjustment will be implemented in a reasonable and consistent manner. The adjustment shall include only the area attributed to the specific target compound. The area adjusted shall not include baseline background noise. The area adjusted shall not extend past the points where the sides of the peak intersect with the baseline noise.

All manually adjusted data shall be clearly identified for approval in the data review process. A permanent record of all manual adjustments shall be maintained in both electronic and hardcopy versions of the data.

Manual adjustment of chromatographic data will not be used to falsify data for any purpose or as a substitute for corrective action on the chromatographic system. Falsification of data through the use of manual peak adjustment is unethical, unlawful and will result in the termination of the offending analyst.

The analyst responsible for changing an integration must date and initial a hard copy of the quantitation report containing and identifying the manual integration.

All manually integrated reported data will be flagged on the raw data, and, when mandated by project-specific requirements, the final data report and associated documentation must provide justification for manual integrations.

Appendix 20.3
ARI's Routine Method 8260B Target Analytes
Internal Standards, Quantitation Ions, and Calibration Criteria

Internal Standard Associated Analyte[1]	CAS Number	Primary Ion	Secondary Ion(s)	Min RRF	Max %RSD	Max %D
Pentafluorobenzene (IS)[1]				--	--	--
Acetone	67-64-1	42	58	0.01	20	20
Acrolein	107-02-8	56	55, 58	0.01	20	20
Acrylonitrile	107-13-1	53	52, 51	0.01	20	20
Bromochloromethane	74-97-5	128	49, 130	0.01	20	20
Bromomethane	74-83-9	94	96	0.01	20	20
2-Butanone	78-93-3	43	57, 72	0.01	20	20
Carbon disulfide	75-15-0	76	78	0.01	20	20
Chloroethane	75-00-3	64	66	0.01	20	20
Chloroform	67-66-3	83	85	0.02	20	20
Chloromethane	74-87-3	50	52	0.01	20	20
Dibromofluoromethane	1868-53-7	111	113	0.01	20	20
Dichlorodifluoromethane	75-71-8	85	87	0.01	20	20
1,1-Dichloroethane	75-34-3	63	65, 83	0.02	20	20
1,1-Dichloroethene	75-35-4	96	61, 98	0.05	20	20
cis-1,2-Dichloroethene	156-59-2	96	61, 98	0.01	20	20
trans-1,2-Dichloroethene	156-60-5	96	61, 98	0.01	20	20
2,2-Dichloropropane	78-87-5	77	97	0.01	20	20
1,1,2-Trichloroethane	79-00-5	97	99	0.01	20	20
Methyl tert butyl ether	1634-04-4	73	57	0.01	20	20
Methylene chloride	75-09-2	84	86, 49	0.01	20	20
1,1,1-Trichloroethane	71-55-6	97	--	0.01	20	20
Trichlorofluoromethane	75-69-4	101	103	0.01	20	20
Vinyl acetate	108-05-4	43	86	0.01	20	20
Vinyl chloride	75-01-4	62	64	0.01	20	20
1,2-Dichloroethane -d4(surrogate	107-06-2	62	98	0.01	20	20
1,1,2-trichloro -1,2,2-trifluoroethane	76-13-1	101	85, 151	0.01	20	20
Iodomethane	74-88-4	142	127	0.01	20	20
Trans-1,4-dichloro-2-butene	110-57-6	53	75	0.01	20	20
Trichloroethene	79-01-6	95	130	0.02	20	20
Chlorobenzene-d5 (IS)	108-90-7	117	82	--	--	--
Chlorodibromomethane	124-48-1	129	127	0.02	20	20
Chlorobenzene	108-90-7	112	77, 114	0.05	20	20
1,3-Dichloropropane	142-28-9	76	78	0.01	20	20
Ethylbenzene	100-41-4	91	106	0.01	20	20

Internal Standard Associated Analyte[1]	CAS Number	Primary Ion	Secondary Ion(s)	Min RRF	Max %RSD	Max %D
2-Hexanone	91-78-6	43	58, 57, 100	0.01	20	20
Styrene	100-42-5	104	78	0.03	20	20
1-chlorohexane	544-10-5	91	55	0.01	20	20
Methylcyclohexane	108-87-2	83	55	0.01	20	20
Cyclohexane	110-82-7	84	56	0.01	20	20
Cyclohexanone	108-94-1	55	42, 98	0.01	20	20
n-Hexane	110-54-3	56	57	0.01	20	20
1,1,1,2-Tetrachloroethane	630-20-6	131	133, 119	0.01	20	20
Tetrachloroethene	127-18-4	166	164, 168, 129	0.02	20	20
M + p-Xylene	108-38-3, 106-42-3	106	91	0.01	20	20
o-Xylene	95-47-6	106	91	0.03	20	20
4-Bromofluorobenzene(surrogate)	400-00-4	95	174	0.01	20	20
1,4-Difluorobenzene (IS)		114	88	0.01	20	20
Benzene	71-43-2	78	52, 77	0.05	20	20
Bromodichloromethane	75-27-4	83	85	0.01	20	20
Toluene-d8(surrogate)	108-88-3	98	100	0.01	20	20
Carbon tetrachloride	56-23-5	117	119	0.01	20	20
2-Chloroethyl vinyl ether	110-75-2	63	65, 106	0.05	20	20
1,2-Dibromoethane	106-93-4	107	109, 188	0.01	20	20
Dibromomethane	74-95-3	93	95, 174	0.01	20	20
1,2-Dichloroethane	107-06-2	62	98	0.01	20	20
1,2-Dichloropropane	78-87-5	63	112	0.01	20	20
1,1-Dichloropropene	563-58-6	75	110, 77	0.01	20	20
cis-1,3-Dichloropropene	10061-01-5	75	77, 110	0.02	20	20
trans-1,3-Dichloropropene	10061-02-6	75	77, 110	0.01	20	20
4-Methyl-2-pentanone	108-10-1	58	43, 100	0.01	20	20
Toluene	108-88-3	92	91	0.04	20	20
1,1,2-trichloroethane	79-00-5	97	--	0.01	20	20
1,4-Dichlorobenzene-d4 (IS)	106-46-7	150	152	0.01	20	20
Bromobenzene	108-86-1	159	77, 158	0.01	20	20
Bromoform	75-25-2	173	175, 254	0.01	20	20
n-Butylbenzene	104-51-4	91	92, 134	0.01	20	20
1,1,2,2-tetrachloroethane	79-34-5	131	133, 119	0.01	20	20
1,2,3-trichloropropane	96-18-4	110	75, 77	0.01	20	20
Trans-1,4-dichloro 2-butene	110-57-6	53	75	0.01	20	20
d4-1,2-Dichlorobenzene(surrogate)	95-50-1	150	152	--	--	--
sec-Butylbenzene	135-98-8	105	134	0.01	20	20
tert-Butylbenzene	98-06-6	119	91, 134	0.01	20	20

Internal Standard Associated Analyte[1]	CAS Number	Primary Ion	Secondary Ion(s)	Min RRF	Max %RSD	Max %D
2-Chlorotoluene	95-49-8	91	126	0.01	20	20
4-Chlorotoluene	106-43-4	91	126	0.01	20	20
1,2-Dibromo-3-chloropropane	96-12-8	75	155, 157	0.005	20	20
1,2-Dichlorobenzene	95-50-1	146	111, 148	0.04	20	20
1,3-Dichlorobenzene	541-73-1	146	111, 148	0.06	20	20
1,4-Dichlorobenzene	106-46-7	146	111, 148	0.05	20	20
Hexachlorobutadiene	87-68-3	225	223, 227	0.01	20	20
Isopropyl benzene	98-82-8	105	120	0.01	20	20
p-Isopropyltoluene	98-87-6	119	134, 91	0.01	20	20
Naphthalene	91-20-3	128	129, 127	0.01	20	20
n-Propylbenzene	103-65-1	91	120	0.01	20	20
1,1,2,2-Tetrachloroethane	79-34-5	83	131, 85	0.03	20	20
1,2,3-Trichlorobenzene	87-61-6	180	145	0.01	20	20
1,2,4-Trichlorobenzene	120-82-1	180	97, 85	0.02	20	20
1,2,3-Trichloropropane	98-18-4	110	75, 77	0.01	20	20
1,2,4-Trimethylbenzene	95-63-6	105	120	0.01	20	20
1,3,5-Trimethylbenzene	95-63-6	105	120		20	20

1 – Compound designations: IS = Internal Standard, SS = Surrogate Standard

Appendix: 20.4
BFB ION ABUNDANCE CRITERIA[1]

Option A: CLP OLM04.2 criteria (default option)‡

BFB MASS INTENSITY SPECIFICATIONS (4-BROMOFLUOROBENZENE)

Mass	Intensity Required (relative abundance)
50	8 to 40% of mass 95
75	30 to 66% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	less than 2% of mass 174
174	50-120% of mass 95
175	4 to 9% of mass 174
176	greater than 93% but less than 101% of mass 174
177	5 to 9% of mass 176

Option B: SW-846 Method 8260C*

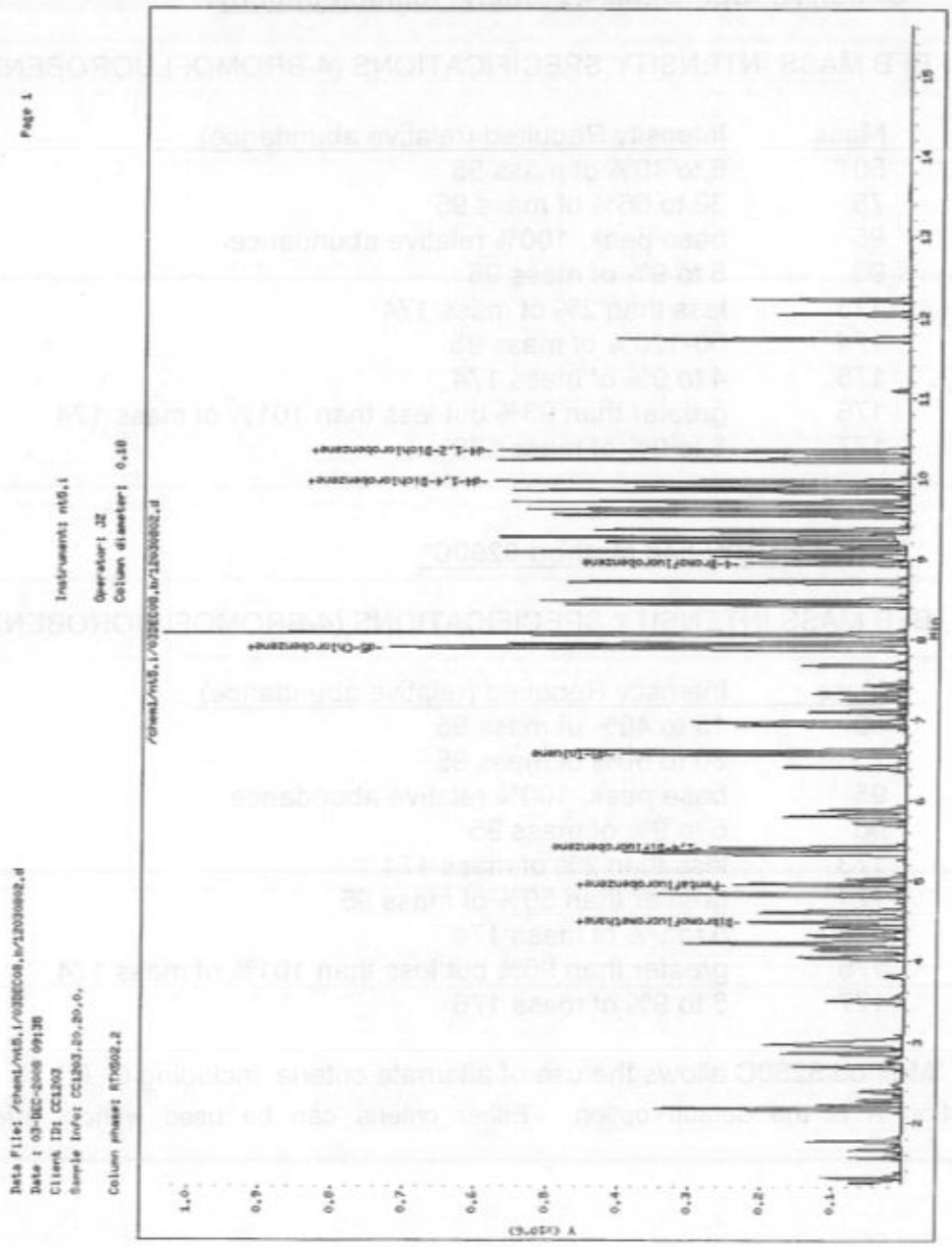
BFB MASS INTENSITY SPECIFICATIONS (4-BROMOFLUOROBENZENE)

Mass	Intensity Required (relative abundance)
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5 to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5 to 9% of mass 176

*Method 8260C allows the use of alternate criteria, including CLP.

‡Option A is the default option. Either criteria can be used without affecting data quality.

Appendix 20.5
Chromatogram of Calibration Standard



Appendix 20.6
GC/MS Volatile Organics Logbook

Analytical Resources Inc.: Organics Instrument Log
NT-3 Serial No.: 10582321978

Date:_____ Analysis: _____ Analyst: _____
GC Program: _____ Column No: _____ Column Type: _____
Instrument Tune (.U or .CT.): _____ EM Voltage: _____
Calibration File: _____ Curve Date: _____

IS/SS	Ical/Ccal	LCS/ICV

Maintenance / Comments

Maintenance Verification (Identify ICal or CCal that demonstrates the instrument is in control):
Every line must contain information or be lined out. Make all entries legible. Start a new page for each QC period.

Form 8038F
Organic Instrument Log

NT-3 12/12/2008
Page 02499

Revision 001
1/16/06

Appendix 20.7
Sample Screening

20.1. Screening of the sample prior to purge-and-trap analysis will provide guidance on whether sample dilution is necessary, and will prevent contamination of the purge-and-trap system.

20.1.1. Aqueous Samples

20.1.1.1. Place an appropriate volume of aqueous sample in a 45 mL vial and fill the vial with organic free water (OFW). Analyze the sample as a normal sample and determine the appropriate dilution for final analysis.

20.1.2. Solid Samples (Soil & Sediment)

20.1.2.1. For screening soils, oils, and solid materials, place 1g of sample into a scintillation vial with 5mL VOA grade Methanol and vortex until well mixed. Dilute 100 µL of the methanol solution into 45 mL OFW. Analyze the sample as a normal sample and determine the appropriate dilution for final analysis.

20.1.3. Waste Samples

20.1.3.1. Water-miscible liquids are analyzed as water samples after first diluting them at least 50 fold with organic-free reagent water.

20.1.3.2. Initial and serial dilutions can be made in a 100mL volumetric flask with organic-free reagent water.

20.1.4. Alternatively, prepare dilutions directly in a 5mL syringe filled with organic-free reagent water by adding at least 180µL, but not more than 900µL, of liquid sample.

20.1.5. OVM may be used to determine approximate dilution levels.

Appendix 20.8
Dry Weight Determination

20.2. Determine the percent dry weight of the soil/sediment sample. Other wastes should be reported on a wet-weight basis.

20.2.1. Weigh 5-10g of the sample into a tared weighing dish.

20.2.2. Place the weighted sample in a 104 ± 2°C oven overnight (12 hour minimum).

20.2.3. Remove the sample from the oven and allow it to equilibrate to ambient temperature.

20.2.4. Calculate the sample per cent dry weight:

$$\% \text{ dry weight} = \frac{\text{sample weight dry (g)}}{\text{sample weight wet (g)}} \times 100$$

Appendix 20.9
Tentatively Identified Compounds

20.3. For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification. Guidelines for making tentative identification are:

20.3.1. Relative intensities of major ions in the reference spectrum (ions >10 % of the most abundant ion) should be present in the sample spectrum.

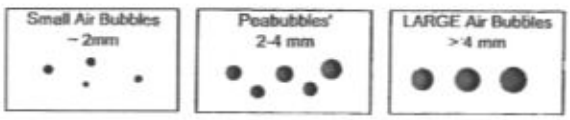
20.3.2. The relative intensities of the major ions should agree within ± 20%.

20.3.3. Molecular ions present in the reference spectrum should be present in the sample spectrum.

20.3.4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

20.3.5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

Appendix 20.9
Pea Size Bubble Reference



Analytical Resources, Incorporated
Analytical Chemists and Consultants

Standard Operating Procedure

Particle Size Distribution
ASTM D421, D422, D2217

SOP 1101
Revision 001

Revision Date: 9/25/05
Effective Date: 4/1/03

Prepared By:

Harold Benny

Approvals:

Harold Benny
Laboratory / Section Manager

David R Mitchell
Quality Assurance

4611 South 134th Place, Suite 100 • Tukwila WA 98168 • **206-695-6200** • **206-695-6201** fax

Annual Review

SOP Number:	1101S
Title:	Particle Size Distribution
Revision:	001
Revision Date:	9/25/05
Effective date:	4/1/03

The ARI employee named below certifies that this SOP is accurate, complete and requires no revisions.

Reviewer's Name	Reviewer's Signature	Date

Analytical Resources, Incorporated
Analytical Chemists and Consultants

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1.0 Scope and Application

This procedure describes methods, materials, equipment, and special conditions required to determine particle size distributions for soil and aggregate. The procedure applies to the determination of particle size distribution required for the classification of materials.

2.0 Materials and Equipment

2.1 BALANCE -- A balance capable of precision to ± 0.01g.
2.2 MECHANICAL SIEVE SHAKER – A mechanical device used to shake and vibrate a nest (stack) of sieves at a constant rate and energy level to separate a given sample into the individual particle sizes.
2.3 SIEVES – A nest of sieves conforming to ASTM E11, typically, a #10, #20, #40, #60, #100, #200, a pan, and a deep #200 (for washing).
2.4 CLOCK – A suitable timing device capable of measurements to the nearest second.
2.5 SEDIMENTATION CYLINDER and RUBBER STOPPER – A standard 1000ml sedimentation cylinder and # 13 rubber stopper.
2.6 HYDROMETER – An ASTM hydrometer, conforming to the requirements of 152H in specification E100.
2.7 STIRRING DEVICE – A standard milkshake mixer type device capable of a speed of not less than 10,000rpm.
2.8 MOTAR and RUBBER TIPPED PESTLE
2.9 TARE DISHES
2.10 SAMPLE SPLITTER
2.11 250ML BEAKERS
2.12 THERMOMETER – Accurate to 0.5°C
2.13 250ml GRADUATED CYLINDER
2.13 1% SOLUTION of SODIUM HEXAMETAPHOSPHATE $Na(PO_2)_6$
2.14 DEIONIZED WATER

3.0 Procedure

3.1 AIR DRIED SAMPLE PREP
3.1.1 Remove samples from storage and verify ID numbers. Notify supervisor of any ID discrepancies. Label and weigh a tare pan for each sample..
3.1.2 Homogenize each sample. Remove the entire sample from container and reduce its size, if necessary, by either quartering or using a sample splitter. Spread the reduced sample to air dry at room temperature. The sample can be mixed periodically during this period to facilitate drying.
3.1.3 When a sample is completely dry, used a mortar and rubber tipped pestle to break up any aggregations.
3.1.4 Approximately 115g is needed for sandy soils and 65g for either silt or clay soils.
3.1.5 Weigh the air-dried sample portion in pan and record the weight on the data sheet as the weight of total sample.
3.1.6 Separate the hydro and sieve sample particles by sieving with a #10 (2.0mm) sieve and collecting the finer particles in a pan.
3.1.7 Grind any portion of sample retained on the #10 sieve with a mortar and rubber tipped pestle until all agglomerations of soil particles are broken into separate grains. Repeat as necessary.
3.1.8 Select about 25g of air-dried sample passing the #10 sieve for moisture content and place in tare dish. Weigh and record weights on data sheet.
3.1.9 Place moisture content sample in the oven set to 105°C and dry to a constant weight, cool in a desiccator, and weigh. Record the weight on data sheet.

3.1.10 Select the portion of ground sample passing the #10 sieve for hydrometer analysis. Tare 250ml beaker, add sample and record weight on data sheet.

3.1.11 Add 125ml Sodium Hexametaphosphate $Na(PO_2)_6$ solution to beaker and stir the sample until thoroughly wetted. Let sample stand for at least 16 hours.

3.1.12 After the soaking period, disperse the sample in the stainless steel "milk shake" mixer. Wash the soil into the cup, using enough deionized water to ensure complete transfer, and to fill the cup at least half full. Run the mixer for 1 minute.

3.1.13 Use lab tape to label 1000ml cylinder with ARI sample number.

3.1.14 Transfer the sample from the mixer cup to the 1000ml cylinder. Rinse the cup thoroughly with deionized water into the cylinder. Fill cylinder to line with deionized water.

3.1.15 If necessary, wash any particles retained in the #10 sieve to remove all remaining fine material.

3.1.16 Place washed #10 materials in oven to dry, and cool in a desiccator, weigh, and record on data sheet.

3.2 WET PREPARATION

3.2.1 Remove sample from refrigerator and check ID numbers. Notify supervisor of any ID discrepancies.

3.2.2 Homogenize sample incorporating any freestanding liquid.

3.2.3 Label and pre-weigh two tare pans, one for moisture content and one for the grain size analysis.

3.2.4 Remove moisture content portion of sample (40-50g) and weigh in tare pan. Record weight on data sheet. Dry sample in oven overnight or until completely dry, cool in desiccator, weigh, and record on data sheet.

3.2.5 Remove grain size portion of sample (approximately 50 to 100 grams based on particles passing the #10 sieve), weigh in tare pan, and record on data sheet.

3.2.6 Label cylinders with sample number by using lab tape.

3.2.7 Place #10 sieve in a pan. Pour the sample onto the sieve and add enough water to cover the sieve by about ½ inch. Aggregated clumps of sediment can be gently broken down with a rubber policeman.

3.2.8 Stir the sample on the sieve with the fingers while gently agitating sieve up and down. Raise the sieve above the water level and complete the washing using a small amount of water. Rinse all remaining material off the #10 sieve back into the tare dish. Place material retained on #10 sieve in oven overnight or until dry (usually 12-16 hours). Put this portion aside to be sieved with rest of sample after hydrometer analysis.

3.2.9 Set aside the pan containing the washings for a period of several hours or until the water above the particles is clear. Decant, pipette, or siphon off as much of the clear water as possible. If the water does not clear up, remove the excess water by evaporation at air temperature, or in an oven at <60C.

3.2.10 Clean workstation when wet sieving is finished. Initial and date data sheet.

3.3 HYDROMETER ANALYSIS

3.3.1 Set up a $Na(PO_2)_6$ blank in a cylinder by using 125ml of $Na(PO_2)_6$ (40 g/L) and bringing it up to the line with deionized water. Set up a rinse cylinder with only deionized water for rinsing the hydrometer.

3.3.2 Take a blank reading by gently lowering the hydrometer in the $Na(PO_2)_6$ solution. The hydrometer should become stable at approximately 5. Read the hydrometer to the nearest whole or half number and record on data sheet. Rinse hydrometer in the plain deionized water cylinder with a gentle "spinning" action. Hydrometer can be left in the rinse cylinder until further use.

3.3.3 Using a rubber stopper, cover the cylinder containing the test sample. Invert the cylinder back and forth for 1 minute (approximately 60 "shakes").

3.3.4 At the end of 1 minute, set the cylinder down and take the temperature of the soil solution and record on the data sheet, then take the first reading at 2 minutes. (If sample becomes "foamy" during shaking see 9.0 CORRECTIVE ACTION.)

3.3.4.1 Hold the hydrometer by the tip. Be sure gloves are dry when handling the hydrometer which can become slippery.

3.3.4.2 Gently lower hydrometer into cylinder. Attempt to "feel" the resistance of the hydrometer being lowered into the sample. At this point gently let go of the hydrometer and let it level. Take the reading to the nearest dash of the hydrometer and record on data sheet. **Do not drop the hydrometer into the sample causing it to dive beneath the surface.**

3.3.4.3 Remove hydrometer slowly from the sample and rinse any sample residue from the hydrometer with a rinse bottle of deionized water into a waste beaker. Rinse the hydrometer in the rinse cylinder with a gentle "spinning" action.

3.3.4.4 Repeat steps 3.3.4.1 to 3.3.4.2 at the following times: 5, 15, 30, 60, 250, and 1440 minutes.

3.3.5 Weigh an empty tare pan and record weight and number on data sheet.

3.3.6 After the last reading, wash cylinder contents through the #200 deep sieve in sink using tap water. Be careful not splash sample outside of sieve. Use a steady stream of water to wash fines through #200 sieve. Rinse sample until water runs clear. Rinse sample into empty tare pan and dry in oven to a constant weight.

3.3.7 Remove sample to be sieved from the oven and cool in desiccator to room temperature. (Combine plus #10 portion at this time if necessary.)

3.3.8 Set up a nest of sieves with the coarsest on the top and grading down to the finest on the bottom over a sieve pan. Clean any dirty sieves with a wire or bristle brush.

3.3.9 Weight sample and tare dish and record on data sheet to the nearest 0.01g.

3.3.10 Add sample to the uppermost sieve in nest. Use brush to clean entire contents of sample from tare dish and to gently break up any agglomerations of material that may have formed due to drying.

3.3.11 Place nest of sieves in mechanical sieve shaker and secure with metal lid with cork right side up. Set shaker arm in down position, set timer for 8 minutes, and start shaker. Close lid. Remove nest when shaker is finished.

3.3.12 Empty top sieve by inverting over a glossy piece of paper. Run bristle brush over bottom of sieve to remove all particles.

3.3.13 Carefully pour sample from wax paper into tare dish, weigh to the nearest 0.01g, and record weight on data sheet.

3.3.14 Repeat 6.4.6 and 6.4.7 with remaining sieves.

3.3.15 Clean workstation when work is finished. Initial and date data sheet.

4.0 CALCULATIONS

The Excel program in the "Template" file under "Regular Hydro" or "Wet Prep Hydro" performs calculations. Refer to ASTM D-422, "Particle Size Analysis of Soils, "Annual Book of ASTM Standards, American Society for Testing and Materials, West Conshohocken, Pennsylvania.

5.0 SAFETY

5.1 LAB WEAR – Lab wear including a lab coat, safety glasses or goggles, and gloves should be worn at all time.

5.2 FINE DUST – Care should be taken not to inhale fine dust while sieving, use a dust mask if necessary. If samples are known to be hazardous, sieving should be done under a hood.
5.3 SIEVE SHAKER – Sieve shaker is loud, and lid should be closed while in operation.
5.4 WORKSTATION – Keep workstation clean at all times. Wipe any spills to avoid safety hazards.

6.0 CORRECTIVE ACTION

6.1 EXCESS WASH VOLUME – If wash volume exceeds 1000ml mark during wet sieving, let sample evaporate until a correct volume is reached.
6.2 FOAMY SAMPLES - Use lab tape (vertically) on cylinder neck to mark chosen whole number on hydrometer if sample is foamy after shaken. After foam has settled re-take hydrometer readings by matching chosen whole number on hydrometer to the marks on tape. Write down hydrometer reading on data sheet.

7.0 REPORT

The report shall include the following:
7.1 Maximum size of particles.
Percentage passing (or retained on) each sieve, which may be tabulated or presented by plotting on a graph.
7.2 Description of sand and gravel particles:
7.2.1 Shape – rounded or angular.
7.2.2 Hardness – hard and durable, soft, or weathered and friable.
7.3 Specific gravity, if unusually high or low.
7.4 Any difficulty in dispersing the fraction passing the No. 10 (2.00-mm) sieve, indicating any change in type and amount of dispersing agent.
7.5 The dispersion device used and the length of the dispersion period.

8.0 REFERENCES

ASTM D-421, "Dry Preparation of Soil Samples for Particle-Size Analysis Determination of Soil Constants, "Annual Book of ASTM Standards, American Society for Testing and Materials, West Conshohocken, Pennsylvania.

ASTM D-422, "Particle Size Analysis of Soils, "Annual Book of ASTM Standards, American Society for Testing and Materials, West Conshohocken, Pennsylvania.

ASTM D-2217, "Wet Preparation of Soil Samples for Particle Size Analysis, "Annual Book of ASTM Standards, American Society for Testing and Materials, West Conshohocken, Pennsylvania.

ASTM E 100, "Hydrometers," Annual Book of ASTM Standards, American Society for Testing and Materials, West Conshohocken, Pennsylvania.

ASTM E 11, "Sieves," Annual Book of ASTM Standards, American Society for Testing and Materials, West Conshohocken, Pennsylvania.

Appendix A – Verification of Sieves

1.0 Purpose
This procedure applies to test sieves used for grain size analysis in the Geotechnical Division lab.

2.0 Frequency
Each sieve shall be visually verified at the beginning of each shift or use. Verification shall be documented every six (6) months.

3.0 Procedure
Each procedure shall be visually inspected to verify that it meets the following requirements.

1. The wire mesh is tight, without distortion or waviness, and there are no punctures or obvious defects in the wire cloth.
2. The joint between the wire cloth and the frame shall be smooth, with no cracks or holes to trap material.
3. Each sieve shall be round and shall easily nest with other sieves.
4. Each sieve shall have a label with the following information
    - U. S. A. standard testing sieve
    - ASTM E-11
    - Standard sieve designation
    - The name of the manufacturer or distributor

4.0 Records
The records generated during the use of this procedure include the Sieve Verification Report, and any nonconformance reports which may result from nonconforming sieves.

Sieve Verification Report

Verification performed by:_____ Procedure No. 1101
Date of verification:_____
Next verification due date:_____

Sieve Number	Frame		Wire Cloth		Joint		Label	
	Accept	Reject	Accept	Reject	Accept	Reject	Accept	Reject

Reviewed and accepted by:_____ Date:_____
Title:_____

Appendix B – Verification of Hydrometers

1.0 PURPOSE

This procedure applies to hydrometers used in the REG Labs. This procedure is to be implemented every six months for the verification of each hydrometer.

2.0 FREQUENCY

Each hydrometer shall be visually verified at the beginning of each shift or use. Verification shall be documented every six (6) months.

3.0 PROCEDURE

Each hydrometer shall be visually inspected to verify that it meets the following requirements.

1. The hydrometer shall be thoroughly dry on the inside when sealed.
2. The hydrometer shall always float with its axis vertical.
3. The glass shall be smooth, transparent and free of bubbles or other imperfections that might interfere with use.
4. Material used for the ballast shall be secured to the lower part of the body, and no loose material of any sort may be inside the hydrometer.
5. Graduation lines and all numbers must be complete and legible.
6. The hydrometer shall be stored in a suitable carton on which shall appear the ASTM number, name, and range.

Hydrometer Verification Report

Calibration: Initial [ ] Routine [ ]

Date of calibration:_____ Performed by:

Next calibration due date:_____Frequency of Calibration: Six (6) months

Hydrometer Number	Dry Inside		Axis		Glass		Ballast		Markings		Carton	
	A	R	A	R	A	R	A	R	A	R	A	R

* A: Accept, R: Reject

Reviewed and accepted by:_____ Date:_____

Title:_____

Analytical Resources, Incorporated
Analytical Chemists and Consultants

Standard Operating Procedure

Moisture Content and Density of Soil
ASTM D2216, D2937

SOP 1103
Revision 001

Revision Date: 9/25/05
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Prepared By:

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Annual Review

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Title: Moisture Content and Density of Soil

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The ARI employee named below certifies that this SOP is accurate, complete and requires no revisions.

Reviewer's Name	Reviewer's Signature	Date

1.0 Scope and Application

This procedure describes the methods, materials, and equipment used to determine the soil moisture content of soils, sediments, rock, and similar materials by mass.

2.0 Equipment

2.1 OVEN – A thermostatically controlled chamber capable of maintaining 110 ±5°C
2.2 BALANCE – A balance capable of precision to ± 0.01g for samples having mass of 200g, and a precision of 0.1g for samples over 200g.
2.3 TARE DISHES
2.4 SPOONS and SPATULAS
2.5 DESICCATOR
2.6 CONTAINER HANDLING APPARATUS – gloves, tongs, or suitable holder for moving and handling hot containers after drying
2.7 SAMPLE EXTRUDER
2.8 CALIPER - The calipers must be capable of measuring with a precision of 0.01cm.

3.0 Procedure

3.1 MOISTURE CONTENT PREP
3.1.1 Remove the samples from storage. Check ID numbers. Notify supervisor of any ID discrepancies.
3.1.2 Label and weigh tare dish and record weight on data sheet.
3.1.3 Homogenize sample, obtain a representative portion of the sample and place it in the tare dish. The representative portion of the sample selected shall be in accordance with the following:

Maximum particle Size (100% passing)	Standard Sieve Size	Recommended minimum mass of moist test specimen for water content reported to ±0.1%	Recommended minimum mass of moist test specimen for water content reported to ±1%
2 mm or less	No. 10	20 g	20g[A]
4.75 mm	No. 4	100 g	20g[A]
9.5 mm	3/8-in.	500 g	50 g
19.0 mm	¾-in.	2.5 kg	250 g
37.5 mm	1 ½ in.	10 kg	1 kg
75.0 mm	3-in.	50 kg	5 kg

[A]To be representative not less than 20 g is used.

3.1.4 For disturbed samples such as trimmings, bag samples, and the like, obtain the test specimen by one of the following methods (listed in order of preference):
3.1.4.1 Thoroughly mix material amenable to manipulation and handling without significant moisture loss or segregation and select a representative portion using a scoop of a size that no more than a few scoopfuls are required to obtain the proper size of specimen defined in 3.1.3.
3.1.4.2 Intact samples such as block, tube, split barrel, and the like, obtain the test specimen by one of the following methods depending on the purpose and potential use of the sample.
3.1.4.3 Using a knife, wire saw, or other sharp cutting device, trim the outside portion of the sample a sufficient distance to see if the material is layered and to remove material that

appears more dry or more wet than the main portion of the sample. If the existence of layering is questionable, slice the sample in half.

3.1.5 Weigh the wet sample and tare and record weight on data sheet.

3.1.6 Place the sample in an oven and dry it to a constant weight (usually 4-24 hours).

3.1.7 Remove from oven and place in a desiccator until cool.

3.1.8 Weigh the dried sample and record dry weight on data sheet.

3.2 WET and DRY DENSITY AND TOTAL POROSITY OF SOILS

3.2.1 Remove the samples from storage. Check ID numbers. Notify the supervisor of any ID discrepancies.

3.2.2 Remove any end caps and inspect the sample. If the sample extends beyond the end of the sleeve, carefully trim the soil until it is equal in length to the sleeve.

3.2.2.1 The sleeve measurements can only be used to determine the volume of the sample provided the sample completely fills the sleeve. If the sleeve is not full, measure the length of the soil in the sleeve for volume determination.

3.2.3 Weigh the sleeve using the following criteria:

3.2.3.1 Length (L), take three separate length measurements at 120 degrees around the perimeter of the sleeve, to the nearest 0.01cm and record on data sheet.

3.2.3.2 Diameter (D), take three separate diameter measurements at 120 degrees around the perimeter of the sleeve, to the nearest 0.01cm and record on data sheet.

3.2.4 Use sample extruder to remove sample from sleeve.

3.2.5 Clean the sleeve and weigh. Record the weight on data sheet to the nearest 0.1g.

4.0 Calculations

4.1 Moisture content (W), as a percent of the dry weight of soil, is calculated as follows:

$$W = [(\text{wet weight of soil} - \text{dry weight of soil}) / \text{dry weight of soil}]) \times 100$$

4.2 Wet and dry density is calculated as follows:

Calculate the wet density by first calculating the total volume of the sample, V_{tot},

$V_{tot} = \pi(D/2)^2 L$

Divide the sample weight in grams by the total volume to get wet density, D_W

D_W = Sample Mass/ V_{tot}

Calculate the dry density, D_D:

$D_D = D_W / (1 + \text{moisture content})$

4.3 Total porosity is calculated as follows:

In order to calculate the total porosity, the specific gravity must either be measured (according to procedure 203), or estimated. The first step is to calculate the volume of the solids:

V_{sol} = Mass of dry solids/specific gravity

Next, calculate the volume of voids by subtracting the volume of solids from the total sample volume:

$V_v = V_{tot}$ – volume of solids

Then divide the volume of voids by the total volume:

$n = V_v/V_{tot}$

5.0 SAFETY

5.1 LAB WEAR – Wearing of personal protective equipment including a lab coat, safety glasses or goggles, and gloves is required at all times.
5.2 WORKSTATION – Keep workstation clean at all times. Wipe any spills to avoid safety hazards.

6.0 REPORT

6.1 Test data forms or test data sheets for MOISTURE CONTENT shall include the following:
6.1.1 Identification of the sample (material) being tested, such as boring number, sample number, test number, container number etc.
6.1.2 Water content of the specimen to the nearest 1% or 0.1%, as appropriate based on the minimum sample used. When using this method in concert with another method, Report the water content of the specimen to the value required by the test method for which the water content is being determined. It may be necessary to use a balance with a greater readability or use a larger specimen mass to obtain the required significant digits the mass of water so that the water content can be determined to the required significant digits.
6.1.3 Indicate if test specimen had a mass less than the minimum indicated in 3.1.3.
6.1.4 Indicate if test specimen contained more than one material type (layered, etc.).
6.1.5 Indicate the temperature of drying if different from 110 ± 5°C.
6.1.6 Indicate if any material (size and amount) excluded from the test specimen.
6.1.7 When reported water content in tables, figures, etc., note any data not meeting the requirements of this test method, such as not meeting the mass, balance, or temperature requirements or a portion of the material is excluded from the test specimen.

7.0 REFERENCES

ASTM D-2216, "Laboratory Determination of Water (Moisture) Content of Soil, Rock, and Soil Aggregate Mixtures", Annual Book of ASTM Standards, American Society of Testing and Materials, Philadelphia, Pennsylvania

ASTM D-2937, "Standard Test Method for Density of Soil in Place by the Drive-Cylinder Method", Annual Book of ASTM Standards, American Society of Testing and Materials, Philadelphia, Pennsylvania

Analytical Resources, Incorporated
Analytical Chemists and Consultants

Standard Operating Procedure

Atterberg Limits Testing of Soils
ASTM D-4318

SOP 1105
Revision 001

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Annual Review

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The ARI employee named below certifies that this SOP is accurate, complete and requires no revisions.

Reviewer's Name	Reviewer's Signature	Date

1.0 PURPOSE AND SCOPE

This procedure describes the methods, materials, equipment, and special conditions required to determine the liquid and plastic limit, and the plasticity index of soils.

2.0 EQUIPMENT

2.1 OVEN – A thermostatically controlled chamber capable of maintaining 110° ± 5° C
2.2 BALANCE – A balance capable of 0.01g with precision to 0.03g
2.3 LIQUID LIMIT DEVICE – A mechanical device consisting of a brass cup suspended from a carriage designed to control its drop onto a hard rubber base. The device may be operated by either a hand crank or electric motor.
2.4 GROOVING TOOL – A tool made of either plastic or non-corroding metal that is used to cut a grove in the soil pat.
2.5 GROUND GLASS PLATE – A ground glass plate at least 30 m square by 1 cm thick for rolling plastic limit threads.
2.6 STORAGE CONTAINER – REGL uses a porcelain bowl and a plastic bag large enough to enclose the dish.
2.7 SIEVES: #10 (2.0MM), #40 (425μm)
2.8 MORTAR and RUBBER TIPPED PESTLE
2.9 DEIONIZED WATER

3.0 DEFINITIONS

3.1 ATTERBERG LIMITS – Originally, six "limits of consistency" of fine-grained soils as defined by Albert Atterberg: The upper limit of viscous flow, the liquid limit, the sticky limit, the cohesion limit, the plastic limit, and the shrinkage limit. In current engineering usage, the term usually refers only to the liquid limit, plastic limit, and in some references, the shrinkage limits.
3.2 LIQUID LIMIT – The water content, in percent, of a soil at the boundary between the liquid and plastic states
3.3 PLASTIC LIMIT – The water content, in percent, of a soil at the boundary between the plastic and semi-solid states
3.4 PLASTICITY INDEX – The range of water content over which a soil behaves plastically. Numerically, it is the difference between the liquid limit and the plastic limit.

4.0 PROCEDURE

4.1 SAMPLE PREP – WET PREPARATION
4.1.1 Remove sample from refrigerator and check ID numbers. Notify supervisor of any ID discrepancies.
4.1.2 Homogenize sample.
4.1.3 Select a portion of sample at natural water content to provide 150 to 200g of material and place in pan or dish. (For #40 wash total amount passing sieve must be 150 to 200g) If by visual examination the soil contains little or no material that would be retained on the #40 sieve, skip to section 4.1.10.
4.1.4 Add enough water to cover the sample and soak until all lumps have softened and the fines no longer adhere to the surfaces of the coarse particles.

4.1.5 Pour the sample-water mixture onto a nest of #10 and #40 sieves place on a pan. Rinse the large particles with a wash bottle to remove the fines and discard coarse material retained on the #10 sieve. Remove the #10 sieve.

4.1.6 Fill the pan holding the #40 sieve with water so that the water level is about 1/2 inch above the screen in the sieve.

4.1.7 Agitate the slurry with fingers and swirl the suspension to wash the fine material from the coarser particles.

4.1.8 Use a wash bottle to rinse the fines from the larger particles and discard the coarse material retained on the #40 sieve.

4.1.9 Reduce the water content of the material passing the #40 sieve until it approaches the liquid limit by air drying, exposing to warm currents (hair dryer), or by decanting clear water off the top. Stir the sample often to prevent over drying of the fringes. **For samples containing soluble salts, do not decant any water.**

4.1.10 Thoroughly mix the sample on the glass plate using a spatula. Adjust the water content of the mixture, if necessary, by adding small increments of distilled water or by air-drying at room temperature while mixing on glass plate. The sample should be at a water content that will result in closure of the groove cut in the sample pat in 20 to 30 blows. (Remove any coarse material found in the sample not washed over #40 sieve.)

4.1.11 Place sample in storage dish and cover to prevent loss of moisture. Allow to stand 16 hours.

4.2 SAMPLE PREP-DRY PREPARATION

4.2.1 Remove samples from refrigerator and check ID numbers. Notify supervisor of any ID discrepancy.

4.2.2 Homogenize sample and select portion to provide 150 to 200 grams of material passing the #40 sieve after processing.

4.2.3 Dry the sample at room temperature until the soil clods pulverize readily.

4.2.4 Using a mortar and rubber-tipped pestle, grind dried sample clods. Remove any concretions, shells, or other fragile particles.

4.2.5 Separate sample on a # 40 sieve. Repeat 4.2.4 with sample remaining on # 40 sieve until all material retained on # 40 sieve consists only of individual sand or grains.

4.2.6 Place material remaining on the # 40 sieve in a dish and soak in a small amount of water. Stir the soil water mixture and pour over # 40 sieve, catching the water and any suspended fines in the washing pan. Add liquid to sample previously sieved on # 40 sieve.

4.2.7 Return to 4.1.9 through 4.1.11.

4.3 LIQUID LIMIT TEST

4.3.1 Thoroughly re-mix the sample before proceeding.

4.3.2 Place a portion of the prepared soil in the cup of the liquid limit device at the point where the cups rests on the base, squeeze it down, and spread it into the cup to a depth of about 10 mm at its deepest point, tapering to form an approximate horizontal surface. Form the pat with as few strokes as possible and to eliminate air bubbles. Keep the unused soil in the storage dish. Cover the storage dish with a plastic bag to retain moisture in the sample.

4.3.3 Beveled edge forward, form a groove in the soil pat by drawing the tool through the soil on a line joining the highest point to the lowest point on the rim of the cup. Hold the grooving tool against the surface of the cup and draw in an "arc", keeping the tool perpendicular to the surface of the cup. In soils where a groove cannot be made in one stroke without tearing the soil, cut the groove with several strokes of the grooving tool or cut the groove slightly less than required dimensions with a spatula and use the grooving tool to bring the groove to final dimensions.

4.3.4 Verify that the base and the underside of cup is clean of debris. Lift and drop the cup by turning the crank at a rate of 1.9 to 2.1 drops per second until the two halves of the soil pat come in contact at the bottom of the groove along a distance of 1/2 inch.

4.3.5 Verify that an air bubble has not caused premature closing of the groove by observing that both sides of the groove have flowed together with approximately the same shape. If this has happened, reform the soil in the cup, adding a small amount of soil to make up for that lost in the grooving operation. Repeat steps 4.3.2 to 4.3.4.

4.3.6 * If the soil slides on the surface of the cup, repeat steps 4.3.1 to 4.3.5 at a higher water content by adding a few drops of water. If, at several trials at successively higher water contents, the pat of soil continues to slide in the cup or if the number of blows required to close the groove is always less than 25, record that the liquid limit could not be determined, and report the soil as non-plastic. It is not necessary at this point to perform the plastic limit test.

4.3.7 Record the number of drops required to close the groove. Remove a slice of soil approximately the width of the spatula, extending from edge to edge of the soil pat at right angles to the groove and including that portion of the groove in which the soil flowed together, place in a pre-weighed container, and cover. If the number of drops is exactly 25 there is no need to repeat the test.

4.3.8 Reform the soil in the cup, adding a small amount of soil to make up for that lost in the grooving operation. Repeat steps 4.3.2 to 4.3.6. If the second closing of the groove requires the same number of drops or no more than two drops difference, remove another water content portion, place in a pre-weighed container. If number of drops is significantly different, add soil remaining in cup to entire sample, remix, and follow steps 4.3.2 to 4.3.6.

4.3.9 Weigh water content portions immediately after completion of the test and record the weight on data sheet.

4.4 PLASTIC LIMIT TEST

4.4.1 Obtain a 20-gram portion of sample from the sample prepared for the liquid limit test.

4.4.2 Reduce the water content of the soil by air-drying or by mixing continuously on the glass plate until it can be rolled without sticking to the hands.

4.4.3 Using 1.5 to 2.0 grams of sample, roll into an ellipsoidal mass.

4.4.4 Roll mass between the palm or fingers and the ground-glass plate with just sufficient pressure to roll the mass into a thread of uniform diameter throughout its length. Continue rolling thread until it is further deformed on each stroke until its diameter equals 1/8 inch and taking no more than 2 minutes. The amount of hand or finger pressure required will vary according to the soil.

4.4.5 When the diameter of the thread becomes 1/8 inch, break the thread into several pieces. Squeeze the pieces together, knead between the thumb and first finger of each hand, reform into an ellipsoidal mass, and re-roll.

4.4.6 Continue 4.4.4 and 4.4.5 until the thread crumbles under the pressure required for rolling and the soil can no longer be rolled into a 1/8-inch diameter thread.

4.4.7 *It has no significance if the thread breaks into threads at shorter length. Roll each of these shorter threads to 1/8-inch diameter. The only requirement for continuing the test is that they are able to be reformed into an ellipsoidal mass and rolled out again.

4.4.8 Place portions of the crumbled threads in a pre-weighed container and cover container.

5.0 CALCULATION

5.1 Calculate the plasticity index as follows:

$$PI = LL - PL$$

where:

LL = liquid limit
PL = plastic limit

6.0 REPORT

6.1 Report the following information:

6.1.1 Any special specimen selection process used, such as removal of sand lenses from undisturbed sample

6.1.2 Sample as air-dried if the sample was air-dried before or during preparation

6.1.3 Liquid limit, plastic limit, and plasticity index to the nearest whole number, omitting the percent designation. When it is not possible to perform liquid limit or plastic limit tests or if the plastic limit is equal to or greater than the liquid limit, report the soil as non-plastic (NP).

6.1.4 The percentage of sample retained on the No. 40 sieve.

6.1.5 The procedure by which liquid limit was performed, if it differs from the multipoint method.

7.0 SAFETY

7.1 LAB WEAR – Wear a lab coat, goggles and gloves at all times.

7.2 WORKSTATION – Keep workstation clean at all times. Wipe any spills to avoid safety hazards.

8.0 REFERENCES

ASTM D-4318, "Liquid Limit, Plastic Limit, and Plasticity Index of Soils, " Annual Book of ASTM STANDARDS, American Society for Testing and Materials, Philadelphia, Pennsylvania

Analytical Resources, Incorporated
Analytical Chemists and Consultants

Standard Operating Procedure

Particle Size Distribution – PSEP Method

SOP 1115
Revision 001

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Annual Review

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The ARI employee named below certifies that this SOP is accurate, complete and requires no revisions.

Reviewer's Name	Reviewer's Signature	Date

1.0 PURPOSE AND SCOPE

This procedure describes methods, materials, equipment, and special conditions required to determine the particle size distribution for sediment samples by the PSEP METHOD. Wet sieving separates sample into two size fractions; particle sizes >#230 sieve and particle sizes <#230 sieve. Fine fractions are further subdivided using a pipetted technique that depends on the differential settling rates of different particles.

Particle size determinations can either include or exclude organic material. If organic material is removed prior to analysis, the "true" (i.e., primarily inorganic) particle size distribution is determined. If the organic material is included in the analysis, the "apparent" (i.e., organic plus inorganic particle size distribution is determined).

2.0 EQUIPMENT

2.1 Balance (Capable of precision to 0.1mg)
2.2 Drying Oven – The oven is thermostatically controlled chamber capable of maintaining a uniform and consistent temperature of 90 ± 2° C.
2.3 Mechanical Sieve Shaker – A mechanical device used to shake and vibrate a nest (stack) of sieves at a constant rate and energy level to separate a given sample into the individual particle sizes.
2.4 Sieves – A nest of sieves including #4 (4.75mm), #10 (2.0mm), #18 (1.0mm), #35 (0.500mm), #60 (0.250mm), #120 (0.125mm) and #230 (.0025).
2.5 Clock – A suitable timing device capable of measurements to the nearest second
2.6 Sedimentation Cylinders and Rubber Stopper
2.7 Desiccator
2.8 25ml in 1/10 Pipette and Rubber Bulb
2.9 Funnel
2.10 Rubber Policeman
2.11 Tare Dishes
2.12 Wire or Bristle Brush and Wax Paper
2.13 Lab Stand with Clamps
2.14 Lab Tape
2.15 Spoons and Spatulas

3.0 REAGENTS

3.1 10% HYDROGEN PEROXIDE (OPTIONAL) – To make 100ml 10% Hydrogen peroxide solution dilute 33.3mls of 30% hydrogen peroxide to 100ml.
3.2 1% Solution of SODIUM HEXAMETAPHOSPHATE $Na(PO_2)_6$ – A batch of 1% solution is made by mixing 40 grams sodium hexametaphosphate $Na(PO_2)_6$ in 1000ml of distilled water. Mix thoroughly and let stand overnight. Acquire batch weight by drying five separate 10ml aliquots in weighed tares overnight or until dry and recording weights to the nearest 0.1mg in batch notebook. Average the five weights and record average in notebook. 1% solution expires in one month.

4.0 DEFINITIONS

4.1 Test Environment – A fairly constant temperature of approximately 20° C during analysis. Small fluctuations in temperature may introduce differences that are of practical significance.
4.2 Sieve Time – Samples will be sieved for 12 minutes.

4.3 Sieves – Sieves are frames that hold wire cloth that has various size openings. The operator will visually examine the sieves for defects (i.e., tears, plugging, holes) prior to each use. Do not use damaged sieves.
4.4 Flocculation – The process where finely suspended particles agglomerate and settle out of solution

5.0 DOCUMENTATION

5.1 PIPETTE GRAIN SIZE ANALYSIS data sheet F101

6.0 PROCEDURE

6.1 Sample Preparation
6.1.1 Remove the samples from the cooler/refrigerator and allow them to warm to room temperature. Verify sample ID numbers. Notify supervisor for ID discrepancies.
6.1.2 To remove organic material from sample see 6.2.
6.1.3 Label and pre-weigh two tare dishes, one for total solids portion of sample and one for wet sieving portion.
6.1.4 Carefully homogenize sample to incorporate any overlying water.
6.1.5 Remove the total solids portion of sample (approximately 25 grams), weigh in labeled tare dish, and record weight on data sheet to the nearest 0.1mg. Dry sample in oven overnight or until completely dry, cool in desiccator, weigh, and record on data sheet.
6.1.6 Remove wet sieve portion of sample (approximately 40 grams), weigh in labeled tare dish, and record weight on data sheet to the nearest 0.1mg. The critical factor for the sample size determination is the weight of the fine-grained material that will be used for the pipette analysis. Ideally, the total dry weight of fine-grained material in the 1000ml-graduated cylinder should equal approximately 15 grams. Estimate fraction of finer than #230 sieve materials along with moisture content (i.e., if the moisture content is 100%, and the percent finer than the #230 is estimated at 50%, an acceptable sample size could be approximately 80 grams).
6.1.7 Clean workstation when sample preparation is finished. Initial and date data sheet.

6.2 Organics Oxidation (optional)
6.2.1 Place sample in large beaker (≥2000ml)
6.2.2 Add 20 ml of 10% hydrogen peroxide solution and mix thoroughly.
6.2.3 Let sample stand until frothing stops.
6.2.4 Add an additional 10ml of 10% hydrogen peroxide solution and mix. Continue adding 10ml portions of solution until no frothing occurs.
6.2.5 Gently boil sample on hotplate to remove excess hydrogen peroxide. Be careful not to lose any material during boiling operation. See section 9.0 CORRECTIVE ACTION if a significant amount of material is lost during boiling operation.

6.3 Wet Sieving
6.3.1 Label cylinders with sample number by using lab tape.
6.3.2 Place a #230 sieve in funnel over 1000ml cylinder using lab stands with clamps. Moisten sieve using a light spray of distilled water.
6.3.3 Add 20-30mls of distilled water in sample tare dish and stir to suspend the fine-grained material.
6.3.4 Pour sample and water onto sieve and gently agitate sieve to separate fractions. Aggregated clumps of sediment can be gently broken down with a rubber policeman. For sample spillage, see 9.0 CORRECTIVE ACTION.

6.3.5 Continue washing sample with a fine spray of distilled water until only clear water passes through sieve. See 9.0 CORRECTIVE ACTION if liquid passes 1000ml mark.
6.3.6 Rinse all remaining material off the #230 sieve back into the tare dish. Place sample in oven overnight or until dry (usually 12-16 hours).
6.3.7 Clean workstation when wet sieving is finished. Initial and date data sheet.

6.4 Sieving the Sand-Gravel Fraction
6.4.1 Remove plus #230 material tare dish from the oven and cool in desiccator to room temperature.
6.4.2 Set up a nest of sieves with the coarsest on the top and grading down to the finest on the bottom over a sieve pan. Clean any dirty sieves with a wire or bristle brush and tap sieve on table with all edges evenly to remove debris.
6.4.3 Weigh sample and tare dish and record on data sheet to the nearest 0.1mg.
6.4.4 Add sample to the uppermost sieve in nest. Use a brush to clean entire contents of sample from tare dish and gently break up any agglomerations of material that may have formed due to drying.
6.4.5 Place nest of sieves in mechanical sieve shaker and secure with metal lid with cork right side up. Set shaker arm in down position, set timer for 12 minutes, and start shaker. Close lid. Remove nest when shaker is finished.
6.4.6 Empty the top sieve by inverting it over a glossy piece of paper. Run bristle brush over bottom of sieve to remove all particles. Tap sieve evenly on table.
6.4.7 Carefully pour sample from wax paper into tare dish, weigh to the nearest 0.1mg, and record weight on data sheet.
6.4.8 Repeat 6.4.6 and 6.4.7 with remaining sieves. Empty material retained in pan to the silt-clay fraction in the cylinder. Compare the total weight retained with the original dry weight to ensure that no material was lost in the sieving process. If weights are significantly different, see section 9.0 CORRECTIVE ACTION. Note large amounts of organic material (e.g., wood debris, grass, shells) or unusual material in any size fraction on data sheet.
6.4.9 Clean workstation when work is finished. Initial and date data sheet.

6.5 Pipetting the Silt-Clay Fraction
6.5.1 Add 10ml of $Na(PO_2)_6$ dispersant to each silt-clay fraction cylinder and fill to the line with distilled water. Record the batch number of dispersant used on data sheet.
6.5.2 Use rubber stopper and mix suspensions by inverting cylinder end over end mixing completely.
6.5.3 Allow the mixed suspension to stand for 2-3 hours and check for signs of flocculation. See 9.0 CORRECTIVE ACTION if flocculation occurs.
6.5.4 Label and pre-weigh all fraction tares to the nearest 0.1mg and record on data sheet.
6.5.5 Print time stickers aliquot withdrawal located at \\Clyde\d\Shared Stuff\Templates\Grain size\PSEP\PSEP Whole on the computer. Attach stickers to the corresponding data sheets.
6.5.6 Use rubber stopper and mix suspensions by inverting cylinder end over end approximately 60 times per one minute. For sample spillage see 9.0 CORRECTIVE ACTION.
6.5.7 After 20 seconds, withdraw a 20ml aliquot from a depth of 20cm below the surface of the suspension using a 25ml 1/10 pipette with rubber bulb. It is critical that the solution be disturbed as little as possible when the pipette aliquots are taken.
6.5.8 Transfer aliquot to pre-weighed tare and rinse pipette by drawing approximately 20ml distilled water into pipette and transferring rinse into the same tare.
6.5.9 Withdraw another 20ml aliquot at the depth of 10cm below the surface of the suspension at the appropriate time as listed in TABLE 1 according to room temperature. For missed pipetting times see 9.0 CORRECTIVE ACTION.

Phi weight = 50 ((E -G) - (F - G))

Where:
E = weight of residue in a 20ml aliquot for a given phi size boundary
F = weight of residue in a 20ml aliquot for next larger phi size boundary
G = weight of dispersant and dissolved salt in a 20ml aliquot

8.0 SAFETY

8.1 Lab wear including a lab coat, safety glasses, and gloves should be worn at all time.
8.2 Care should be taken not to inhale fine dust while sieving. A dust mask should be worn when sieving.
8.3 The sieve shaker is loud, and the lid should be closed while in operation.
8.4 Keep workstation clean at all times. Wipe any spills to avoid safety hazards.

9.0 CORRECTIVE ACTION

9.1 When sample is lost during organics oxidation notify the laboatory supervisor. A significant loss of sample may require re-analysis.
9.2 Sample loss during wet sieving - If sample is spilled on table use distilled water to wash spillage into tare dish. If sample is lost on the floor, see supervisor. A significant loss may result in a redo.
9.3 Excess wash volume – If wash volume exceeds 1000ml mark during wet sieving, let sample evaporate to obtain an acceptable volume.
9.4 Sample loss during sieving – Attempt to brush spilled sample into tare dish prior to weighing. It is extremely important to keep worktables and floor clean prior to sieving in case a spill occurs.
9.5 Sample Flocculation – Flocculation results in a curdling and rapid settling of lumps of particles or by the presence of a thick, soupy layer on the bottom of the cylinder passing abruptly into clear water above. When flocculation occurs, add dispersant in 10ml increments until no noticeable flocculation is observed. Record the total volume of dispersant added on the data sheet.
9.6 Sample spillage during cylinder mixing – If rubber stopper is not tight on cylinder and spillage occurs, continue pipetting procedure. Note approximate amount of spilled liquid and note on data sheet.
9.7 Missed pipetting aliquots – If withdrawal is missed the suspension may be re-mixed and the missed aliquot can be taken at the appropriate time. It is not necessary to take the initial 20ml aliquot for this corrective action.

10.0 REFERENCES

10.1 Folk, Robert L., 1978, The Petrology of Sedimentary Rocks, Hemophile Publishing Co., Austin TX
10.2 USACOE 1995, Puget Sound Estuary Protocols, U.S. Army Corps of Engineers, Seattle WA

Analytical Resources, Incorporated
Analytical Chemists and Consultants

Standard Operating Procedure

Moisture and Organic Content of Soil
ASTM D2974

SOP 1145
Revision 001

Revision Date: 9/25/05
Effective Date: 4/1/03

Prepared By:

Harold Benny

Approvals:

Harold Benny
Laboratory / Section Manager

David R Mitchell
Quality Assurance

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6.5.10 Dry all aliquots in oven to a constant weight at 90°C.
6.5.11 Cool dried samples to room temperature in a desiccator, weigh to the nearest 0.1mg, and record on data sheet.
6.5.12 Keep workstation clean. Initial and date data sheet.

TABLE 1. Withdrawal Times for Pipette Samples

Diameter Finer (phi)	Diameter Finer (um)	Withdrawal Depth (cm)	Elapsed Time for Withdrawal of Sample in Hours (h) Minutes (m) and Seconds (s)						
			18°	19°	20°	21°	22°	23°	24°
4.0	62.5	20	20s	20s	20s	20s	20s	20s	20s
5.0	31.2	10	2m	1m 57s	1m 54s	1m 51s	1m 49s	1m 46s	1m 44s
6.0	15.6	10	8m	7m 48s	7m 36s	7m 25s	7m 15s	7m 5s	6m 55s
7.0	7.8	10	31m 9s	31m 11s	30m 26s	29m 41s	28m 59s	28 m 18s	27 m 39s
8.0	3.9	10	2h 8m	2h 5m	2h 2m	1h 59m	1h 56m	1h 53 m	1h 51 m
9.0	1.95	10	8h 32m	8h 18m	8h 6m	7h 56m	7h 44m	7h 32 m	7h 22 m
10.0	0.98	10	34h 6m	33h 16m	32h 28m	31h 40m	30h 56m	30h 12 m	29h 30 m

7.0 CALCULATIONS

7.1 MOISTURE CONTENT and TOTAL SOLIDS

Total solids content is determined as follows:

$$\text{Percent Solids} = \frac{(A - B)(100)}{(C - B)}$$

Where: A = weight of tare and dry sample residue
B = weight of tare
C = weight of tare and wet sample

7.2 SAND and GRAVEL FRACTION

The sand and gravel fractions of the sample are reduced as follows:

$$\text{Percent retained (for a given sieve)} = C/D * 100$$

Where: C = cumulative weight retained for a given sieve
D = total dry sample weight

7.3 SILT and CLAY FRACTION

The total weight of the phi-sized interval in the 1000ml graduated cylinder is determined as follows:

Phi weight = 50 ((E -G) - (F - G))

Where:
E = weight of residue in a 20ml aliquot for a given phi size boundary
F = weight of residue in a 20ml aliquot for next larger phi size boundary
G = weight of dispersant and dissolved salt in a 20ml aliquot

8.0 SAFETY

8.1 Lab wear including a lab coat, safety glasses, and gloves should be worn at all time.
8.2 Care should be taken not to inhale fine dust while sieving. A dust mask should be worn when sieving.
8.3 The sieve shaker is loud, and the lid should be closed while in operation.
8.4 Keep workstation clean at all times. Wipe any spills to avoid safety hazards.

9.0 CORRECTIVE ACTION

9.1 When sample is lost during organics oxidation notify the laboatory supervisor. A significant loss of sample may require re-analysis.
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Annual Review

SOP Number:	1145S
Title:	Moisture and Organic Content of Soil
Revision:	001
Revision Date:	9/25/05
Effective date:	4/1/03

The ARI employee named below certifies that this SOP is accurate, complete and requires no revisions.

Reviewer's Name	Reviewer's Signature	Date

1.0 PURPOSE AND SCOPE

This procedure describes the methods, materials, equipment, and special conditions required to determine ash content and organic matter in peats, clays, silts, and muck.

2.0 EQUIPMENT

2.1 Balance – A balance with precision to 0.01g
2.2 Drying Oven – A thermostatically controlled chamber capable of maintaining a temperature of 100° ± 5°C.
2.3 Muffle Furnace – A furnace capable of a constant temperature of 440°- 750°C.
2.4 Evaporating Dishes – Use porcelain bowls. Ignite clean evaporating dishes at 440 ± 10° C for 1 hour in muffle furnace to remove any remaining organic material.
2.5 Spoons and Spatulas
2.6 Furnace Togs
2.7 Sample Splitter
2.8 Oven Gloves

3.0 PROCEDURE

3.1 Turn on Furnace and set to correct temperature (440°).
3.2 Remove sample from refrigerator and check client ID numbers. Notify supervisor of any ID discrepancies. Allow samples to warm to room temperature.
3.3 Label and pre-weigh evaporating dish. Record the information on a data sheet.
3.4 Homogenize sample. Remove portion of sample (for WDOE modified method, at least 300g) and place in evaporating dish. Weigh sample and record the wet weight on the data sheet.
3.5 Place sample in oven and dry to a constant weight, cool in desiccator, weigh and record dry sample weight on data sheet.
3.6 Return sample to oven and dry to a constant weight, cool in desiccator, weight and record dry sample weight on data sheet
3.7 Place evaporating dish and sample in muffle furnace and hold until sample is completely ashed (times vary according to organic content in samples).
3.8 Remove sample from furnace using furnace tongs and place on ceramic plate until sample cools enough to handle with oven mitt. Place sample in desiccator until cooled to room temperature (see 6.0 CORRECTIVE ACTION for sample spills). Record the ashed weight on the data sheet.
3.9 Return evaporating dish and sample to the muffle furnace and hold for at least 2 hours, or longer if necessary.
3.10 Remove sample from furnace using furnace tongs and place on ceramic plate until sample cools enough to handle with oven mitt. Place sample in desiccator until cooled to room temperature. Record the ashed weight on the data sheet.
3.11 If sample sets out for a period of time, place in drying oven to insure constant weight, cool in desiccator, weigh and record the ashed weight on data sheet.

4.0 CALCULATIONS

4.1 To calculate moisture content (W):

$$W = [(\text{wet weight of soil} - \text{dry weight of soil}) / \text{dry weight of soil})] \times 100$$

4.2 To calculate ash content:

Ash = [(Dry soil - ashed soil) / Dry soil] x 100

4.3 To calculate organic content:

Organic Content = [(Dry weight - ashed weight) / Dry weight] x 100

5.0 SAFETY

5.1 Lab wear including lab coat, safety glasses or goggles, and gloves should be worn at all times.
5.2 Keep workstation clean at all times. Wipe any spills to avoid safety hazards.
5.3 Use oven gloves when removing samples from oven. Bowls will be hot.
5.4 Use furnace tongs and oven gloves to remove samples from muffle furnace. Sample bowls will become extremely hot. Setting hot sample bowls on plastic or other soft surface will cause damage to counters. Do not handle sample bowl with oven gloves until it has cooled.

6.0 CORRECTIVE ACTION

6.1 SPILLED SAMPLE – If sample is spilled in any process of this procedure, attempt to brush the sample back into the bowl. Samples containing flammable materials may "pop" and scatter material inside the muffle furnace. It is extremely important to always keep ovens and furnaces clean in order to recover spilled samples. If the sample cannot be recovered, notify your supervisor.

7.0 REPORT

7.1 Results for organic matter and ash content, to the nearest 0.1%.
7.2 Furnace temperature used for ash content determinations
7.3 Whether moisture contents are by proportion of as-received mass or oven-dried mass
7.3.1 Express results for moisture content as a percentage of as-received mass to the nearest 0.1%.
7.3.2 Express results for moisture content as a percentage of oven-dried mass as follows:
7.3.2.1 Below 100% to the nearest 1%
7.3.2.2 Between 100% and 500% to the nearest 5%
7.3.2.3 Between 500% and 1000% to the nearest 10%
7.3.2.4 Above 1000% to the nearest 20%

8.0 REFERENCES

ASTM D-2216, "Laboratory Determination of Water (Moisture) Content of Soil, Rock, and Soil Aggregate Mixtures", Annual Book of ASTM Standards, American Society of Testing and Materials, Philadelphia, Pennsylvania

ASTM D-2974, "Standard Test Methods for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils", Annual Book of ASTM Standards, American Society of Testing and Materials, Philadelphia, PA

STANDARD OPERATING PROCEDURES

Pore Water Sampling Using a PushPoint Mini-Piezometer

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STANDARD OPERATING PROCEDURES
Pore Water Sampling Using a PushPoint Mini-Piezometer

1.0 GENERAL

This standard operating procedure (SOP) outlines the methods and procedures for sampling interstitial pore water from the transition zone of permeable sediments using a mini-piezometer device. The detailed procedures are based on the use of the PushPoint sampler designed by MHE Products (http://www-personal.engin.umich.edu/~markhen/downloads.html); however, the procedures outlined are applicable to similar piezometer sampling devices.

The PushPoint sampler consists of a small diameter Type 316 stainless steel tube screened at one end and with a sampling port at the other end. The screened area consists of a series of machine cut slots presenting approximately 20% open area. The screened area is limited to the section near the tip. A guard rod provides structural support to the sampler and prevents the sampler from being plugged or bent by the sediment or debris on insertion. Following insertion of the sampler to the desired depth the guard rod is removed and suction is applied to the sample port to develop pore water flow and for sampling.

2.0 SAMPLER INSTALLATION METHODS

This section outlines detailed procedures for installing the samplers in the transition zone of the target sediments. The PushPoint sampler is a reusable device provided the interior bore of the sampler is kept clean and the body of the sampler is protected from being bent. The screened area near the tip is easily deformed if the guard rod is not inserted before inserting the device into the sediment or removing the device. The machined tolerances between the sampling tube and the guard rod can be excessively worn if grit is trapped in the bore. The guard rod may also become jammed if silt is trapped in the damaged bore.

Sampling in marine or tidally influenced systems is usually conducted on a falling tide before the low tide when groundwater discharge is expected to be the greatest. Sampling in non-tidal systems is usually scheduled during periods of peak groundwater flow. In shallow water the samplers can be deployed from a boat or by a person while wading.

Insertion of the PushPoint sampler into the sediment should only be done with the guard rod fully inserted into the bore. The sampler is inserted into the sediment with a twisting motion until the specified sampling depth (e.g., 1 ft or 30 cm) is reached or until refusal. If samples are collected at multiple depths in a single hole the samples should be collected from the deepest to the shallowest to

eliminate the necessity to insert the sampler deeper into the sediment once the guard rod is removed. If multiple samples are taken at one hole or if lateral movement will enlarge the sampler hole then the use of a large flat sampling plate with a supporting hole through the center can be used to stabilize the sample tube.

Once the sampler is inserted to the specified depth the guard rod is removed and the piezometer is developed. Tubing is attached to the sampling port at the top of the sampler and a gentle suction is applied using either a syringe or a peristaltic pump. Water is withdrawn from the sampler at a low flow rate (recommended: 50 to 200 milliliters per minute [mL/minute]). The initial volume of turbid water is usually 20 to 50 mL depending on the size of the sampler and the sediment type. Once the water clears representative samples of the pore water may be collected. If the sampler is plugged flow rates may be insufficient for sampling. A syringe can be used to apply a vacuum that is rapidly released sending a pressure pulse through the sampler and increasing flow rates.

The establishment of proper pore water flow through the sampler can be confirmed by monitoring various water quality parameters such as turbidity, temperature, conductivity, pH, and dissolved oxygen. These water quality parameters should be monitored over time until they stabilize.

3.0 SAMPLE COLLECTION METHODS

This section describes procedures for sample collection, handling, and processing. Collection of representative samples of the pore water requires the proper development of pore water flow through the piezometer. Sampling flow rates during development should be low enough that turbidity is minimal and that overlying surface water is not drawn into the sampler.

Discrete water samples can be collected using a syringe. Pore water samples collected in syringes can be labeled and placed on ice for later transfer to appropriate sample containers. Pore water samples can also be collected using a peristaltic pump. Samples from a peristaltic pump can be transferred directly into appropriate sample containers. All sample containers must be labeled with the collection site, appropriate collection information, and include the proposed analyte list.

Syringes used for collection of pore water for volatile organic compound (VOC) analysis should be constructed of 100% polyethylene/ polypropylene. Samples for VOC analysis should have minimum exposure to the air. Transfer of the pore water from the syringe to the sample containers requires using a piece of tubing to overfill the container from the bottom of the container with a minimum of splashing. Sample bottles may also be filled with the peristaltic pump by overfilling the container with the tubing under the water surface in the container to displace all the air.

4.0 EQUIPMENT CLEANING, DECONTAMINATION

Sample containers, instruments, working surfaces, technician protective gear, and other items that may come into contact with sediment sample material must meet high standards of cleanliness. Sample containers will be provided by the laboratory and will be precleaned, certified, and individually labeled with a lot number traceable to a Certificate of Analysis.

All samplers and sample collection equipment will be cleaned and decontaminated prior to arrival at the site. The Geomatrix standard decontamination procedure for the PushPoint sampler, tubing, pumps, and other sample handling equipment is modeled after Puget Sound Estuary Program (PSEP) protocols (PSEP, 1997); however, the decontamination procedure will not use any acid or solvent rinses (the final rinse will use distilled water). The decontamination procedure is as follows:

1. Prewash rinse with tap water.
2. First wash with solution of tap water and Alconox soap (brush).
3. Second rinse with tap water.
4. Second wash with solution of tap water and Alconox soap (brush).
5. Final rinse with tap water.
6. Final rinse with distilled water.
7. Coverage (no contact) of all decontaminated items with aluminum foil.
8. Storage in clean, closed container prior to use.

All wash and rinse solutions will be collected and disposed of properly.

5.0 SAMPLE DESIGNATIONS

Each sample collected will be given a unique sample identification number. Sample identification numbers will include the site name (former Rhone Poulenc or RP), the sample date (mmddyy), and a sample sequence number. For example, a sample ID of RP081411-03 would identify the third sample collected on 08/14/11. The sampling sequence number will not include the monitoring well number or indicators of field blanks, equipment blanks, etc. A master sampling log that documents the sequence numbers and the corresponding wells will be maintained by the field personnel in accordance with the existing hydraulic control interim measures groundwater sampling Quality Assurance Project Plan (URS, 2002).

6.0 SAMPLE HANDLING

Pore water samples will be kept in sight of the sampling crew or in a secure, locked vehicle at all times. Samples will be transported to the office at the end of the day for storage (samples will be placed in coolers with ice or in a refrigerator) until transferred to the testing laboratories. Transfer of samples from the field crew custody to the laboratory custody will be documented using chain-of-custody procedures. If someone other than the sample collector transports samples to the laboratory, the collector will sign and date the chain-of-custody form and insert the name of the person or firm transporting the samples under "transported by" before sealing the container with a Custody Seal.

Samples not scheduled for the initial analysis round will be archived and stored at the analytical laboratory in a secure area.

7.0 FIELD QUALITY CONTROL REQUIREMENTS

Data and log forms produced in the field will be reviewed daily by the person recording the data, so that any errors or omissions can be corrected. All completed data sheets will be removed daily from the field clipboard and photocopied; the original data sheets will be filed in a fireproof file cabinet and the photocopies stored in the project file. All data transcribed from field forms into electronic forms and tables will be 100 percent verified for accuracy and freedom from transcription errors.

8.0 WASTE MANAGEMENT

All waste derived during this investigation will be placed in proper containers, labeled, characterized, and disposed of in accordance with the appropriate regulations.

9.0 REFERENCES

PSEP (Puget Sound Estuary Program), 1997, Recommended Guidelines for Sampling Marine Sediment, Water Column, and Tissue in Puget Sound: Prepared for the U.S. Environmental Protection Agency and Puget Sound Water Quality Action Team.

URS (URS Corporation), 2002, Interim Measures Performance Monitoring Plan, Quality Assurance Project Plan: Prepared for Container Properties, L.L.C., September.